Microstructure of Steel- cement Paste Contact Zone in Concrete Modified with Mineral Additives and Chemical Admixtures

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1. Introduction

Until now much attention has been paid to cement paste and aggregate contact zone[5, 7]. In general terms it can be said that the microstructure of hydrated cement paste in direct contact with coarse aggregate differs from the microstructure of the internal layers of cement paste. The contact zone near the aggregate has much bigger porosity than the hydrated cement paste some distance away from the aggregate. For that reason, microscraches are initiated in this layer, which negatively affects the resistance of the cement composite. Both coarse and fine aggregate make up irregular inclusions in the cement matrix and weaken the resistance of the composite. Another additional element in the cement matrix are steel rods used for the construction reinforcement. It should be assumed that by analogy to the coarse aggregate, a regular weakened contact zone is also created around the steel (in a form of steel fibers, 1mm in diameter, dispersed in the matrix or steel rods placed in concrete, 24 mm in diameter), located along the rods in the whole circuit[1, 2, 4, 6].
In the construction concrete, steel rods located in the stretching zone are exposed to the influence of very high contact forces causing coagulation between cement paste and steel. The safety line (for stability loss) of the reinforced concrete element depends on external load which has to be transferred by the concrete in the compression zone, and in the stretching zone by steel rods. Steel rods work effectively until the contact zone becomes destroyed between the cement paste and concrete.
For that reason, it is extremely important to investigate this phenomenon which has not been as widely described in literature as the contact zone of cement paste and aggregate.

2. Research description

The research was divided into two parts. In the first part, the adhesive forces between steel rods and modified cement matrix were determined empirically. In the second part, microstructures of cement paste and steel contact zone were determined by means of scanning microscopy.

2.1. Testing adhesive force between cement paste and steel rods

In order to prepare cement paste, we used Portland cement commonly produced in Poland– both ordinary type and containing some additives, as well as special HSR cement. Mineral composition of the used cement types are presented in Table 1.

Table 1 Mineral composition of cement types used in tests

Kind of cement	Mineral composition [%]			
	C_3S	C_2S	C_3A	C_4AF
CEM I 32,5	63,72	12,78	7,71	7,44
CEM I 32,5R	60,94	16,11	7,69	6,87
CEM II/B-SV 32,5	38,08	14,14	6,08	4,26
CEM III/A 32,5 NA	29,29	10,88	4,68	3,28
CEM I 42,5 HSR NA	58,80	16,40	2,73	14,59

Ultimate compressive strength of these cements after 7, 14, 28, 60 and 90 days of curing was compared and presented in Fig. 1.
These cements show diverse ultimate compressive strength. Cements: CEM I, CEM II and CEM III show comparable compression strength, CEM I R and CEM I HSR are characterized by much higher ultimate compressive strength.
From the above mentioned binding materials, standard cement paste with w/c = 0.5 and non-standard paste with w/c = 1 were made. Standard cement pastes made of CEM I were additionally modified, reducing water volume and maintaining standard consistency.
Modification was made using the following:

- superplasticizer FM 1.8% m.c. and silica fume SF 10% m.c.,
- polymer emulsion BD10 15% m.c.,
- NITCAL^{TM} 290 m.c. admixture consisting of $Ca(NO_3)_2$ – 79%, NH_4NO_3 – 6%, H_2O – 15%.

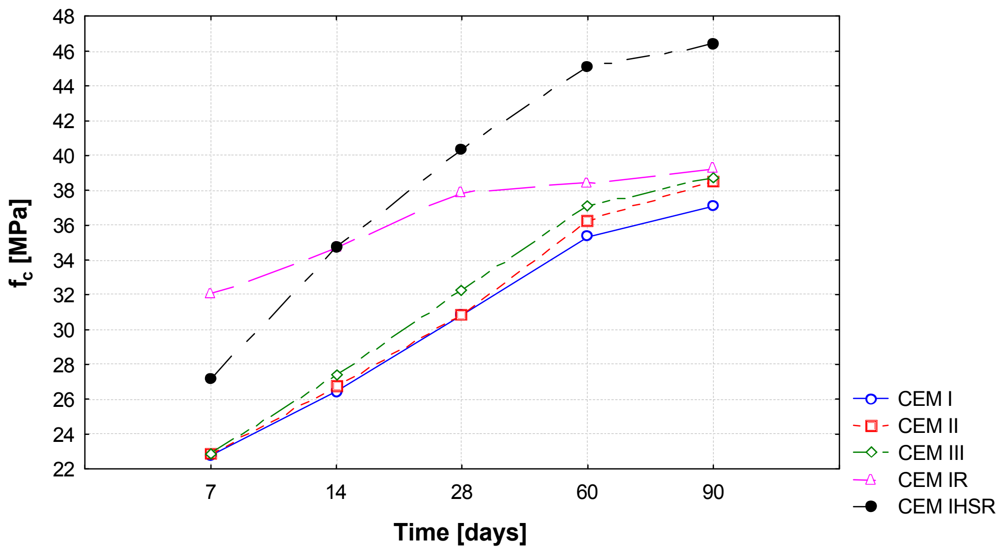

Fig. 1 Ultimate compressive strength of standard cement paste made of various cement types

Ordinary cement paste made of CEM I with w/c = 1 was used to achieve a porous and absorbable cement paste. Altogether, 17 types of cement paste were prepared.

In order to determine the adhesive forces, 17 series of cylindrical cement samples were made, 75 mm in diameter and 200 mm in height, with a 10 mm in diameter steel rod inside. Such a prepared sample was placed in a testing machine on a test stand . Clock extensometer registered protruding of the rod and the machine reader registered the value of the force causing the rod's movement.

Maximum force causing the loss of adherence between the steel rod and cement paste as well as protruding of the steel rod were tested.

2.2. Testing steel-cement paste contact zone

Contact zone was assessed by means of a scanning microscope JSM 5500 LV with an available accelerating voltage of 0.3 - 20 kV, 50 - 300000 fold magnification, 10 nm resolution and maximum sample sizes of 100x25x25 mm. Due to such small sizes of samples, their cutting from reinforced concrete was given up for the sake of specially prepared cylindrical samples. Empty, previously cleaned and etched steel sleeves were used - 15 mm in diameter, 40 mm high with a notch in the middle of the height. The sleeves were filled in with cement paste with composition as in point 2.1 and after achieving 28 day resistance they were broken in liquid nitrogen and placed on the microscope table.

3. Test results of adherence forces between steel rods and modified cement matrix

Force measurement results (kN) causing the loss of adherence of steel rods to ordinary cement matrix and cement matrix modified with chemical admixtures and mineral additives are listed in Fig. 2. The tests were carried out for three CEM I cements.

Regardless of cement type, the adherence of steel to concrete was similar in all cases. The highest forces (180 kN) causing the loss of steel adherence to cement paste occurred when the matrix was modified with super plasticizer and silica fume. Steel rods in cement paste with addition of polymer dispersion and calcium nitrate showed good adherence. Lower adherence was recorded in case of steel rods placed in ordinary cement paste (105 kN) and very low force (30 kN) was recorded when rods were placed in a porous cement paste with w/c = 1.0.

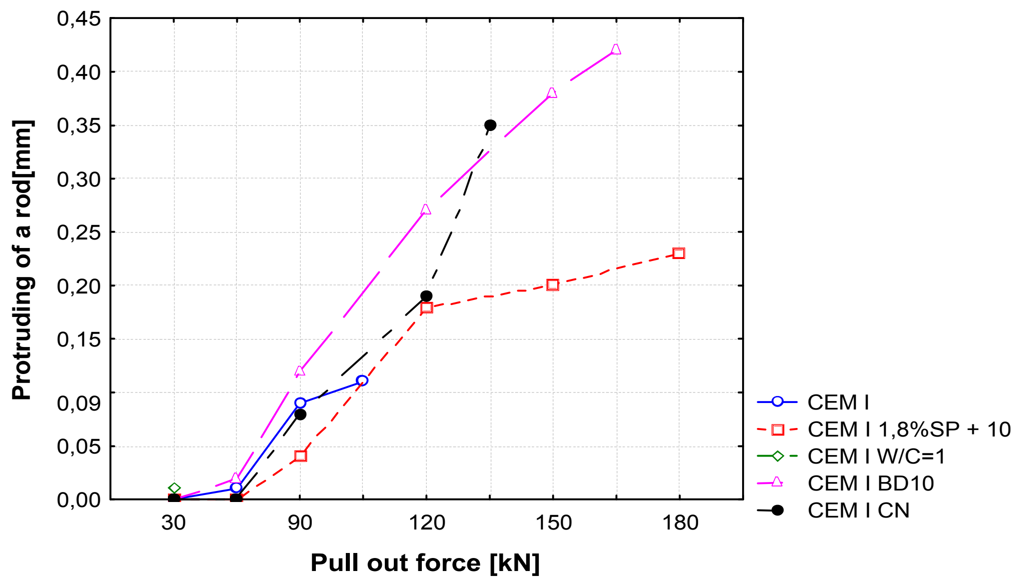

Fig. 2 Ratio of pull-out force and rod protruding for various modifications of Portland cement CEM I 42,5HSR.

In Fig. 2 one more dependence can be noticed. Even though cement paste with SP and SF(silica fume) achieved the best adherence results, protruding of the steel rod preceding the loss of adherence is not big when compared to other cement pastes (0,25 mm).
This means that the density of the structure in direct contact zone of cement paste and steel is highest from all tested modified pastes. In case of using polymer emulsion, the situation looks different. The pull-out force affecting the adherence is slightly lower but the steel and cement paste connection is more flexible as the loss of contact occurs when dislocation is 0,45 mm.

4. Microscopic test results

Scanning photos of 17 types of cement paste were taken according to the procedure described in point 2.2. When the photos were being taken, some problems occurred with simultaneous observation of steel and concrete due to different contrast of both materials .Due to this, the next photos show contact layers of steel and cement paste but with no image of steel.

In the initial test phase, the review of microstructure of standard paste of 4 cement types was made – figure 3 a, b, c, d . In case of ordinary Portland cement (CEM I fig. 3 a) ettringite and $Ca(OH)_2$ crystals can be noticed. In the ettringite environment, hydrated phases of CSH gel can be observed. In SV cement images, apart from a marginal quantity of CSH gel, we can also see grains of fly ashes filling the microstructure. CEM III/A cement creates a different image of cement paste. On hydrated $Ca(OH)_2$ crystals there is a large amount of ettringite originating from the aluminum included in slag. The smallest amount of ettringite occurs in pastes containing HSR cement. This is justified as the cement includes slight volumes of C_3A in comparison with other ordinary Portland cements.

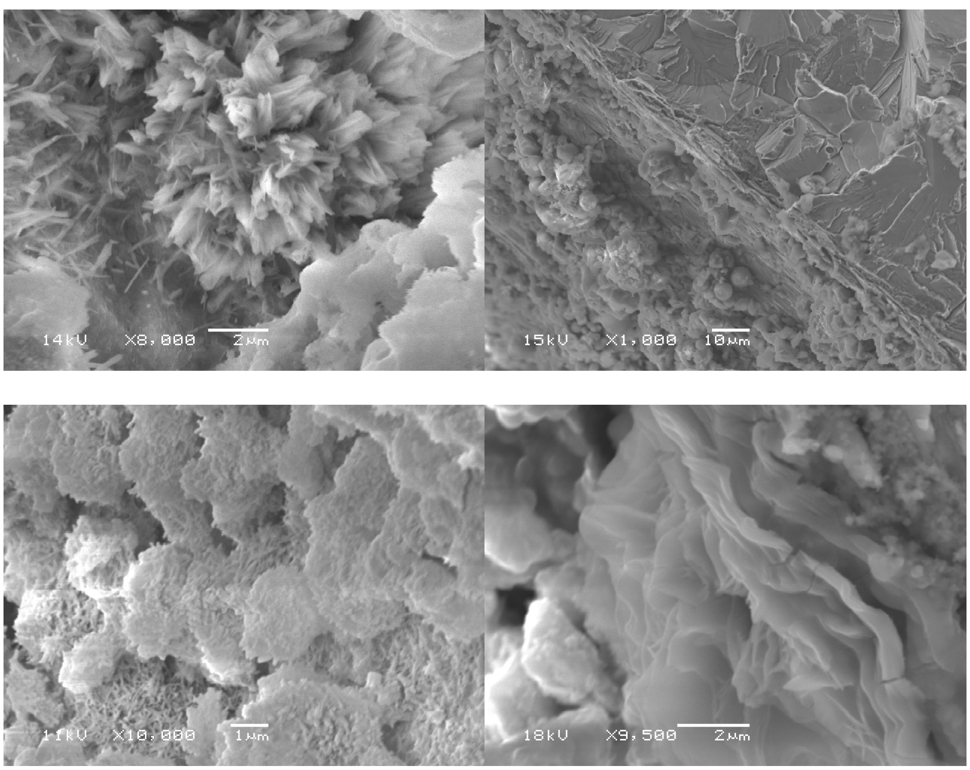

Fig. 3 a, b, c, d Contact zone for : a - CEM I 32,5, b – CEM II/B, c – CEM III/A, d- CEM I 42,5 HSR

The next set of pictures shows the images of microstructure of steel – modified paste 1.8% SP + 10% SF contact zone. Lower volume of the make-up water, while maintaining the desired consistency, caused the creation of smaller air pores after the hydration of components (Fig. 4). Concentration of the microstructure by hydrates with silica fume improves the contact zone. Silica gel causing the increase of matrix resistance also has an impact on the increase of adherence forces, which was demonstrated by measurements presented in Fig. 2.

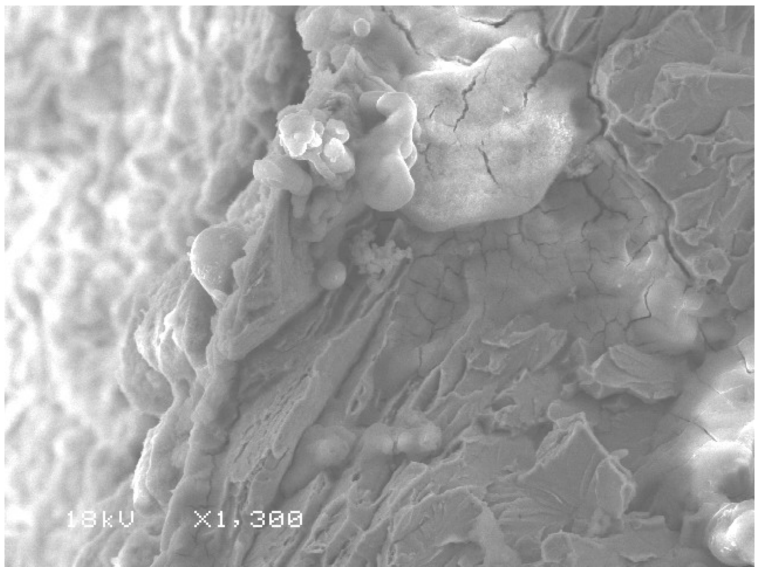

Fig. 4 Contact zone for CEM I 42,5 HSR with SP + SF

Different image of contact zone microstructure was observed in case of the cement paste modified with polymer emulsion BD10. Clearly visible polymer fibers with length ranging from 5 to 25 μm, located incidentally, penetrate CSH gel element (Fig. 5). The reduction of make-up water volume causes such concentration of microstructure that the share of pores adhering directly to the steel is quite low. Mutual cooperation of fibers and hydrated calcium silicates lead to good adherence of steel to the paste (CSH impact) and improved flexibility of this connection (BD 10). In scanning images we can also see $Ca(OH)_2$ crystals. Their location 16μm from the contact zone has no impact on this connection.

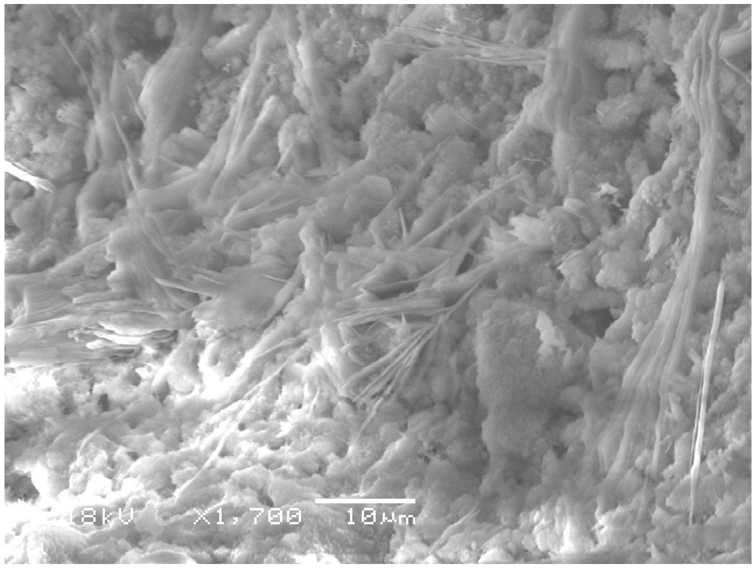

Fig. 5. Contact zone for CEM I 32,5 with polymer additive BD10

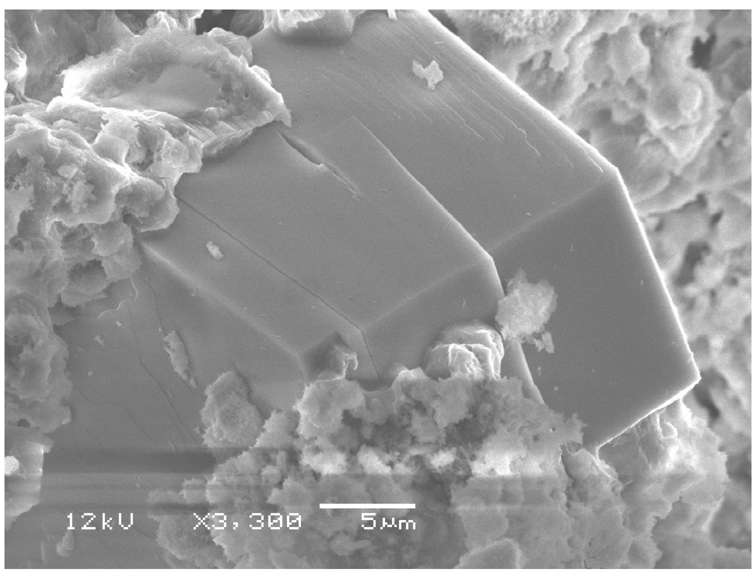

Fig. 6 Contact zone for CEM I 32,5 R with calcium nitrate admixture

The highest concentration of the microstructure could be observed in the cement paste with calcium nitrate (Fig. 6). Big clusters of CSH and immersed in them hexagonal plates of calcium hydroxide create a compact microstructure devoid of air pores. This explains good adherence of steel to cement paste with calcium nitrate admixture (Fig. 2) but not as good as in case of SF additive.

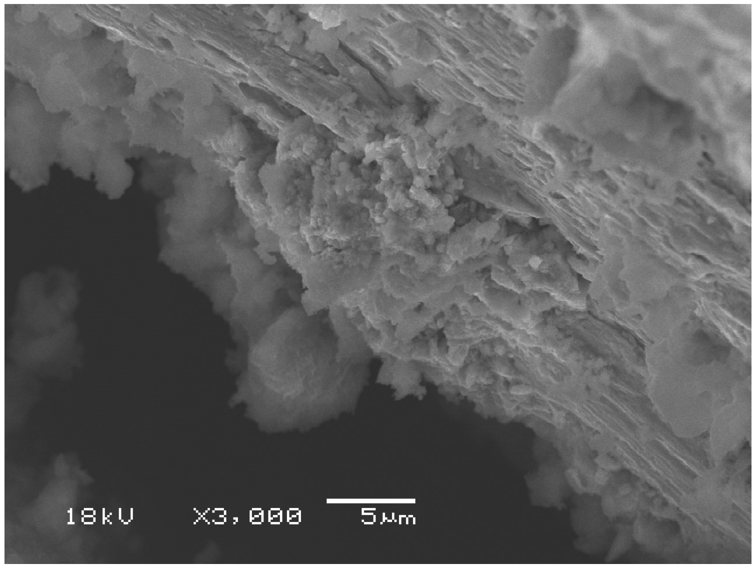

Fig. 7 Contact zone for w/c = 1 mortar with CEM I 32,5

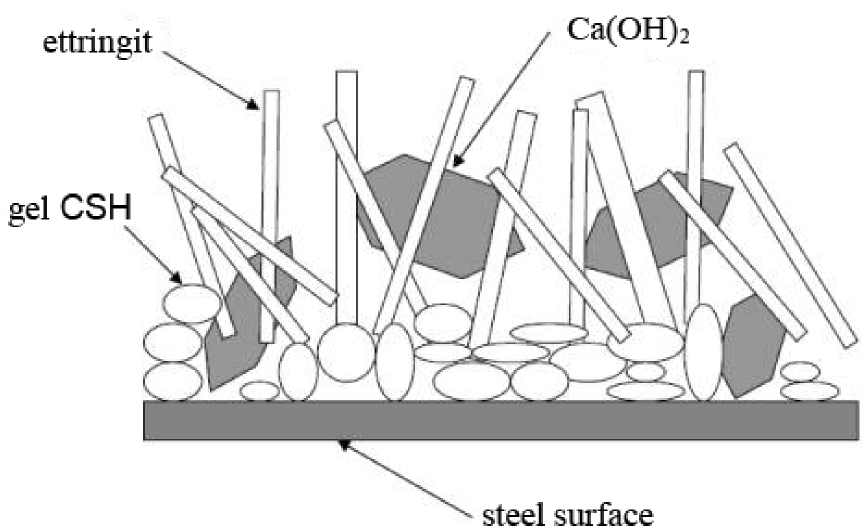

Fig. 8 General pattern of contact zone between cement paste and steel rebar

Samples of paste with w/c = 1.0 presented in Fig. 7 show high porosity of microstructure. Such a low concentration of the structure results in low adherence of steel to cement paste. In the photos we can see well hydrated grains of Portland cement and small gel clusters in the empty space between steel and concrete. Such contact enables migration of aggressive particles and destruction of the composite. The presented scanning tests of contact layer microstructure allow the presentation of a general pattern of contact zone (Fig. 8).

5. Conclusions

Direct contact zone of steel and cement paste as a component of the mortar has a different character than the microstructure of the mortar itself. Apart from the separation of some components (CSH gel, polymer fiber, Portland cement) and their concentration on the rod's surface we can observe the creation of overlapping or penetrating layers in the area of 40 – 50 μm from the reinforcement.

Contact zone of steel and cement paste also looks different than the contact zone of aggregate and cement paste presented for instance by Zimbelmann [7]. For the contact of cement paste – aggregate, the first contact layer is ettringite, for the contact of steel – cement paste, according to the tests made by the authors, in most cases it is CSH gel.

The performed tests are an expansion of the border zone of steel and concrete presented by A. Bentur, S. Diamond and S. Mindess in 1985 [3]. According to these authors, the border layer is enriched with plate crystals of Portland cement, whose "c" axis is perpendicular to the reinforcement surface. This layer is not continuous and in the created gaps etringit as well as particles of CSH phase can be found – sometimes surrounding Portland cement crystals. Microstructure of border zone is changeable and depends on cement and reinforcement types, on additives, conditions of concrete curing and other factors. Tests carried out by the authors of this report are the confirmation of the impact of various factors on the steel-cement paste contact layer.

6. Acknowledgements

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7. References

[1] Al. Khalaf, C.L. Page: Steel-mortar interfaces : microstructure features and mode of failure, Cement and Concrete Research,1979, vol. 9, p. 197-207

[2] D. Barnes, S. Diamond, W. L. Dolch: The contact zone between Portland cement paste and glass “aggregate”surfaces, Cement and Concrete Research, 1978, vol.8, p. 233-243,

[3] Bentur, S. Diamond, S. Mindess: Cracking processes in steel fiber reinforced cement paste, Cement and Concrete Research, 1985, vol. 15, p. 331- 342,

[4] E. Gjorv, P. J. M. Monteneiro, P. K. Mechta: Effect of condensed silica fume on the steel concrete Bond, ACI Materials Journal, 1990, vol. 87, No. 6, p. 573 – 585,

[5] M. Neville: Properties of concrete, Fourth edition, London 1995, pp. 844

[6] J. Pinchin, D. Tabor: Interfacial phenomena in steel fibre reinforced cement : Structure and strength of interfacial region, Cement ad Concrete Research, 1978, vol. 8, p. 15 – 24,

[7] R. Zimbelmann: A contribution to the problem of cement – aggregate bond, Cement and Concrete Research, 1985, vol. 15, p. 801- 808.