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Microstructure and Mechanical Properties of a Steel – Cement Mortar Interface Zone in Concrete Modified with Mineral Additives and Chemical Admixtures

Jozef Jasiczak ¹, Mariusz Januszewski ²

¹ Institute of Structural Engineering, Poznan University of Technology, Piotrowo 5, 60-965 Poznan, Poland

² Division of Civil Engineering and Building Materials, Koszalin University of Technology, Sniadeckich 2, 75-453 Koszalin, Poland

jozef.jasiczak@put.poznan.pl

Abstract. The Zimbelmann's model presenting the interface layer of aggregate and cement paste is often published in technical literature. It is also known as the border zone of steel fibre and cement concrete presented by A. Bentur, S. Diamond, S. Mindess in 1985, and W. Kurdowski in 1991. The authors of this article have conducted a wide range of research regarding the contact layer of steel and cement mortar in reinforced concrete. Fifteen composition variants with various cements have been analysed, including those modified with additives and chemical admixtures. Adhesive forces of mortar to steel, as well as the contact microstructure have been specified. The microstructure was tested on specially prepared samples made of steel pipes filled with cement mortar. Fractures of the samples were prepared in a liquid nitrogen atmosphere and the contact of steel and paste was estimated under a scanning microscope at a magnification of 1000-10000. A contemporary model of cement interface was developed, consisting of a preliminary phase of CSH, crystalline phase of $\text{Ca}(\text{OH})_2$ and ettringite phase. The pull on strength, thickness of CSH layer and its porosity depend on the composition of cement in a specific phase and the use of w/c additives and admixtures.

1. Introduction

A lot of attention has been given so far to the cement grout – aggregate interface zone [1, 2]. In general, it can be stated that the microstructure of hydrated cement grout in direct vicinity of coarse aggregate grain, differs from the microstructure of external grout layers. The interface by the aggregate is much more porous than hydrated cement grout, remote from coarse aggregate. For this reason, microscratches are initiated in this layer, which significantly weakens the strength of a cement composite. Both coarse and fine aggregate are irregular inclusions in a cement matrix and spatially weaken the strength of the composite. Another filler of a cement matrix are steel reinforcement bars. By analogy to coarse aggregate, we should assume that a regular, weakened transition zone located along the bar, around its entire perimeter, is formed around steel (in the form of steel fibres with a diameter up to 1 mm, distributed in a matrix or concreted steel bars, with a diameter up to 24 mm) [3, 4, 5].

The structural concrete steel bars located in the tensile zone, as well as the compressed zone are exposed to very large static forces, inducing shear between the cement grout and steel. The safety limit (loss of stability) of a reinforced concrete element depends on the magnitude of external loads, which must be transferred within the compressed zone by concrete and steel bars, and in the tensile zone by



steel bars. Steel bars are effective for as long as the interface zone between the cement grout and concrete is not destroyed.

However, the steel-concrete interface zone is determined not only by mechanical features of concrete and steel, but also by the nature of the mortar, shaped by the impact of chemical admixtures and mineral additives on the concrete [6, 7, 8].

Therefore, it is extremely important to understand the phenomenon, which has been given much less attention in the literature than the grout-aggregate.

2. Research methodology

2.1. Mortar variants adopted for the study

Standard Portland cements CEM I 32.5 and CEM I 32.5 R and sulphate-resistant cement CEM I 42.5 HSR mass-produced in Poland were used to prepare the cement mortars.

Cement phase composition, calculated with the Bogue method, is given in Table 1.

Table 1. Phase composition of cements, %

Type of cement	Cement phases			
	C ³ S	C ² S	C ³ A	C ⁴ AF
CEM I 32.5	64	13	8	7
CEM I 32.5R	62	16	8	7
CEM I 42.5 HSR NA	59	16	3	15

Compressive strength of these cements after 7, 14, 28, 60 and 90 days of curing is shown in figure 1.

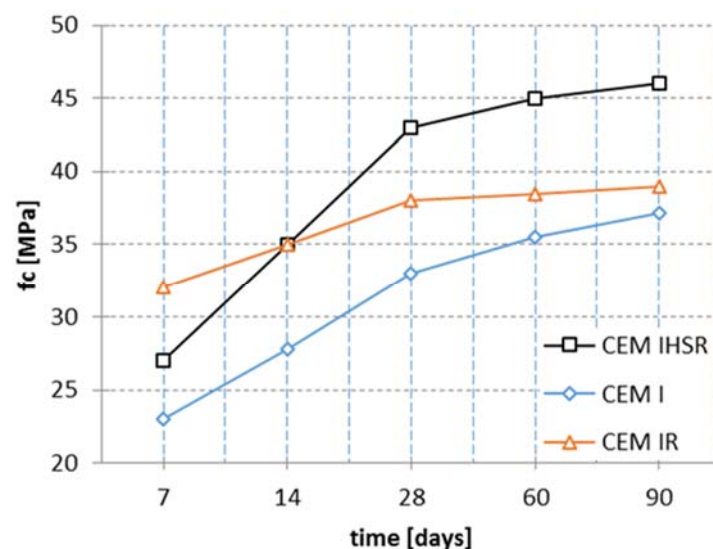


Figure 1. Compressive strength after 7, 14, 28, 60, 90 days of 3 cement standard mortars

Each cement type was used to prepare five standard mortars and five mortars modified acc. to the recipes shown in Table 2.

A total of 15 types of grout, with variable strength properties, in the range of 20 - 45 MPa, after 28 days of curing were prepared.

Table 2. Composition of mortars adopted for the study [g/dm³]

Recipes	Mortar designation, g/dm ³				
	Z1	Z2	Z3	Z4	Z5
Cement	450	450	450	450	450
Aggregate	1350	1350	1350	1350	1350
Water	225	180	450	157.5	225
Polymer emulsion, 15% t.w.	-	-	-	67.5	-
NITCAL™, 2% t.w.	-	-	-	-	9
Superplasticizer, 1.8% t.w.	-	8.1	-	-	-
Silica fume, 10% t.w.	-	45	-	-	-
W/C Ratio	0.5	0.4	1.0	0.35	0.5

2.2. Studies regarding the forces of adhesion of mortar to smooth steel bars

The “pull-on” method (pressing a steel bar into concrete) was adopted for the determination of adhesive forces instead of the “pull-off” method (pulling a steel bar out of concrete).

The shearing force values obtained with both methods were compared in paper [9]. By assuming the pressing of a bar, data on shear stresses at the material boundaries in reinforced concrete compressed structures are obtained. In order to determine the adhesive forces of a smooth steel bar to grout, 15 series (3 pcs in each) of cement cylindrical samples with a diameter of 75 mm and a height of 200 mm, and a $\phi 10$ mm steel bar inside were prepared. The measurements were taken at a stand shown in Figure 2.

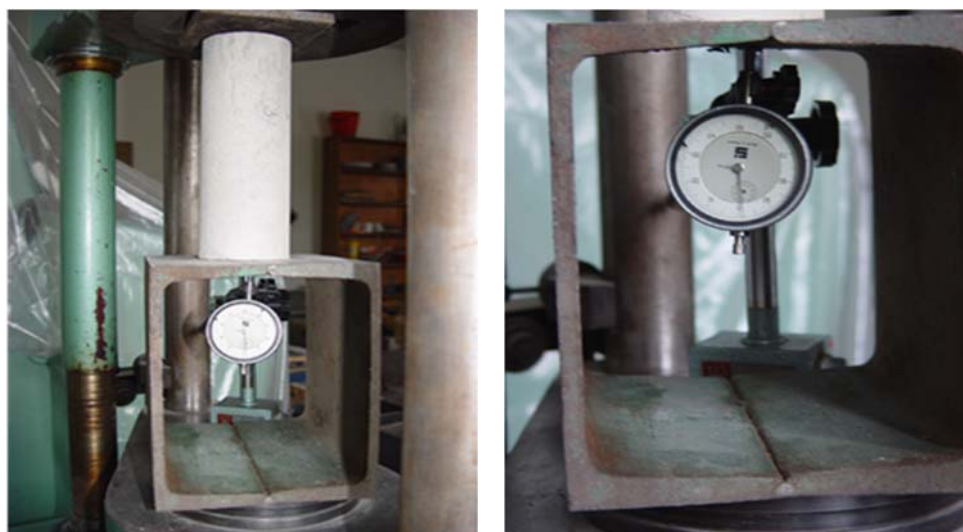


Figure 2. Test stand for measuring adhesive forces between grout and a steel bar; a – general view, b – displacement measurement sensor

Dial strain gauge at the bottom of the sample recorded bar retraction, while the reader of the tester recorded the force causing the displacement. The tests include the maximum and proportional to the displacements force causing loss of adhesion between a steel bar and the grout, as well as the pull-off increments of a steel bar.

2.3. Studies involving the images of a steel-cement grout interface zone

The interface zone images were evaluated with the use of a JSM 5500 LV scanning microscope, with an available accelerating voltage of 0.3÷20 kV, 50÷300000 zoom, resolution of 10 nm and maximum sample dimensions of 100×25×25 mm. For these reasons, it was decided not to cut samples out of an

aforementioned cylindrical sample with a steel bar inside, in favour of specially prepared cylindrical samples.

Empty, previously cleaned and etched steel sleeves, with a diameter of 15 mm and a height of 40 mm, with a notch at half height were used. Sleeves were filled with mortar as shown in point 2.1, and after obtaining a 28-day strength, they were fractured in a liquid nitrogen atmosphere and placed on a microscope observation stage. Samples and details of the steel-mortar contact are shown in Figure 3.



Figure 3. Sleeves filled with mortar after fracturing. Notch edge in the top part of the sample can be seen on the photo. Contact magnification on the adjacent photo

Due to different steel colours (silver colour) and cement mortar (shades of grey), the lens of the microscope was pointed at the external surface of a metal tube, with making an SEM of a cement matrix and an image of the steel surface in the background. Diversified SEM images were obtained, depending on the manner of mortar modification.

3. Test result of steel bars to modified cement matrix adhesive force

The results of measuring forces (kN) causing loss of adhesion of steel bars to a standard cement matrix and a matrix modified with chemical admixtures and mineral additives are listed in Figure 4. The tests were conducted for three CEM cements (CEM I 32.5; CEM I 32.5 R; CEM I 42.5HSR) and five material variants.

Regardless of the type of cement, the adhesion of steel to concrete was similar in all the three cases, and the differences concern only the values of the steel bar displacements. Considerable differences can be observed between individual material variants of the mortars. The largest forces (180 kN) causing full loss of steel-mortar adhesion appear when modifying a matrix with a superplasticizer and silica fume. Good adhesion (135, 150/165 kN) is exhibited by steel bars placed in a mortar with added calcium nitrite and polymer dispersion. Lower adhesion is exhibited by steel bars placed in standard cement mortar (force 105 kN), and minor (30 kN) by bars placed in porous cement mortar with $w/c = 1.0$.

Figure 4 additionally indicates other significant relationships. In each case, apart from the mortar with $w/c = 1$, it is possible to distinguish a proportional increase of the force to vertical displacement of the bar in the initial image of the graph. Papers [8, 10] state that at this stage of the displacements, we are dealing with initial adhesion resulting from chemical bonds of concrete and steel. After “breaking” of these bonds, we are dealing only with friction of the bar and adjoining hydrates against the cement matrix, hence, the force increments cause disproportionate bar displacement values, until it is protruded.

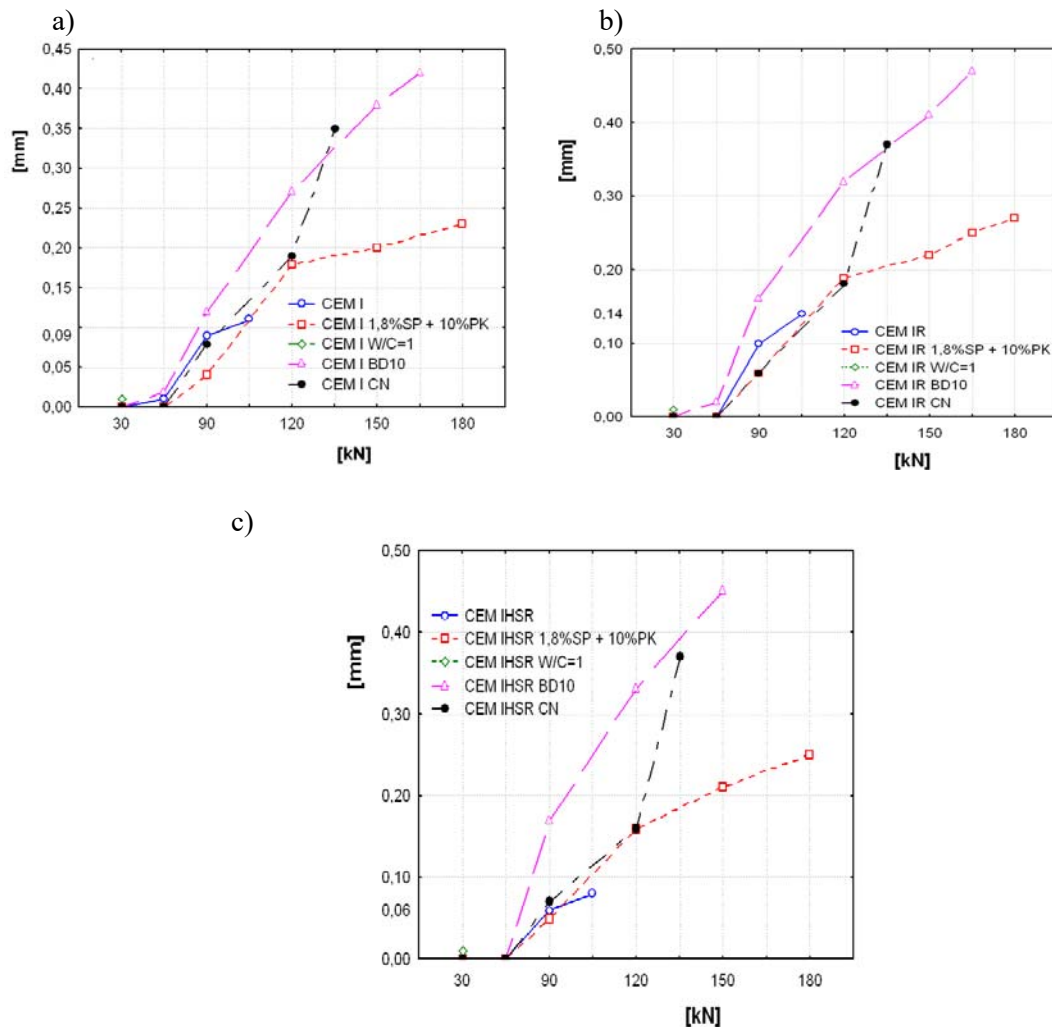


Figure 4. The value of the “pull-on” force causing and increment of bar pull-out for different modification of cement mortars: a) CEM I 32.5; b) CEM I 32.5 R; c) CEM I 42.5HSR

The determination of both values, converted to tangential stresses τ_p calculated from the relationship is extremely useful when dimensioning reinforced concrete structures subject to compression. The mentioned relationships for CEM I 32.5 N cement mortar are shown in Table 3, and the results are commented in the conclusions. Considerable differences concern the adhesive forces associated with the modifications of the standard mortar.

$$\tau_p = P / 2\pi r l, \quad \text{N/mm}^2 \text{ [MPa]} \quad (1)$$

where:

- P – destructive force in kN, taken from the graphs in Figure 4,
- $2\pi r l$ – sheared side surface of a $\phi 10$ mm and 200 mm long steel bar (6280 mm^2)

From Figure 4, you can additionally read one more relation. While the mortar with SP and SF achieved the best strength results, the displacement of a steel bar preceded by adhesion loss is not large in this case (0.25 mm), compared to other mortars. It means that the density of the structure in the direct grout-steel contact zone is the largest among the tested modified mortars. In the case of using polymer emulsion, the situation is different. The adhesion breaking force is in this case slightly lower, however,

the steel-mortar connection more flexible, because loss of contact takes place at a displacement of 0.42 mm. A similar comment can be made in regard to the mortar with added NITCALTM.

Table 3. The values of displacements and shear stresses on the steel-concrete interface in reinforced concrete for the mortar with CEM I 32.5 N

Designation	Standard mortar	Mortar +1.8% SP+ 10% SF	Mortar w/c=1	Mortar + polymer emulsion	Mortar + NITCAL TM
$\tau_{p \text{ chem.}}$	14.33	19.11	4.78	14.33	19.11
Δl	0.085	0.17	0.01	0.12	0.19
$\tau_{p \text{ max}}$	16.72	28.66	4.78	26.27	21.5
Δl_{max}	0.10	0.23	0.01	0.42	0.35

4. Results of microscopic tests

Scanning photos, according to the procedure described in point 2.3, were taken for 15 mortar types. Microphotographs of mainly cement hydrates were taken, showing only a side view of the steel surface (Figure 5). It was observed that the steel and mortar interface zone was continuous neither in a macro, nor in a micro scale. Layer contact in a micro scale may be of a more local (point) and layered nature than continuous, along the entire mortar-steel contact surface, and the gaps between the layers may be 5 - 10 μm . Other microphotographs are shown in Figure 6a, b, c, d. The images differ in terms of density of the interface transition layer. The most compact is the transition layer from CEM I 42.5 HSR cement. The nature of this layer is also similar in the other modifications. The interface zone of layers modified with 1.8% SP + 10% SF exhibits the greatest compactness and continuity of all the cases, as well as thickness reaching 8 μm in some places (Figure 6a). Also, the values of shear stresses transferred by the transition zone are the biggest for these cases (cf. Figure 4). The interface layers in a mortar with w/c = 1 adjoining steel are very thin, and the distances from parallel layers are several μm from the first one (Figure 6b). For this reason, steel-concrete adhesion exhibits the lower values. Matrices modified with polymer emulsion present a very interesting image (Figure 6c). We can see short polymer fibres, 5 to 25 μm long penetrating the CSH gel elements, making the matrix elastic, and, at the same time, during steel bar displacement, constituting a type of bonds, which are broken only upon a bar displacement of 0.45 mm (only 0.25mm in case of a matrix with 1.8 % SP + 10 % SF).

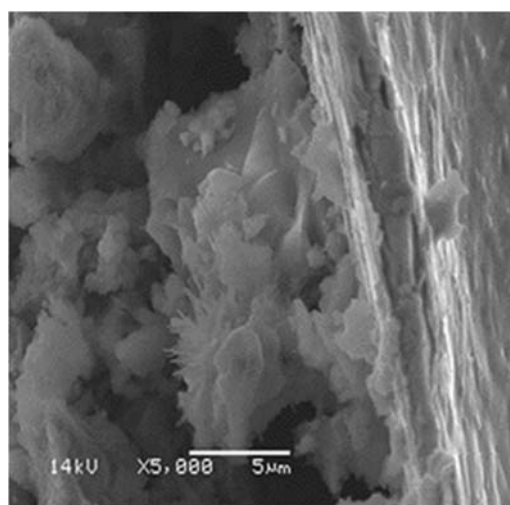


Figure 5. Photo arrangement: standard mortar: (right) steel surface, (centre and left) hydrates with a discontinuous contact nature

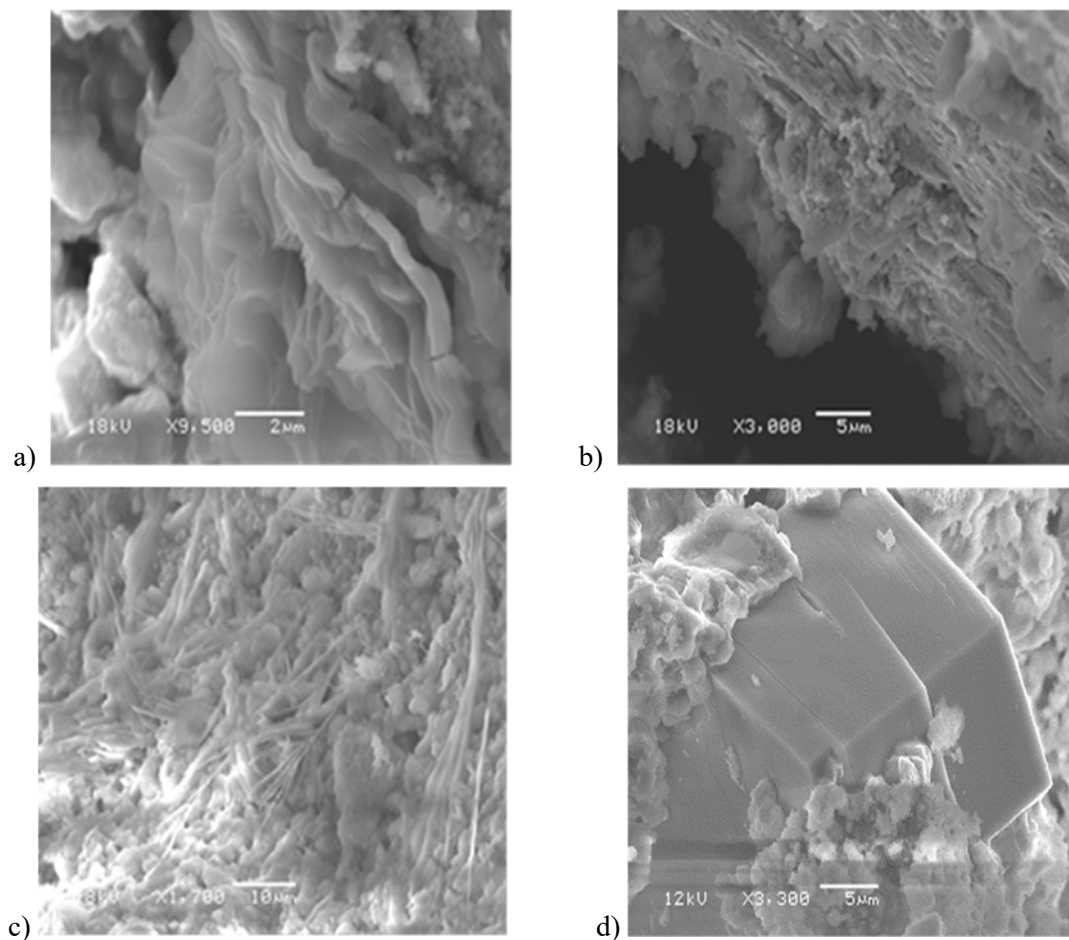


Figure 6. Images of cement matrices adjoining steel surface: a) mortar with 1.8% SP + 10% SF, b) mortar with $w/c = 1$; gaps in lagging; c) mortar with polymer emulsion, d) mortar with NITCALTM admixture

An example of good structural density are also mortars with an admixture of NITCALTM (Figure 6 d). CSH clusters and hexagonal portlandite plates parallel to the steel surface. (which is also noted by W. Kurdowski in the paper [11]), form a compact microstructure. It explains the reasons for the high adhesion of steel to mortars with added NITCALTM, however, not as high, as when adding 1.8 % SP + 10 % SF.

5. Conclusions

The presented research by the authors indicate that the interface zone between steel and cement grout, which is a mortar ingredient, is of different nature than the microstructure of the mortar itself. The made modifications also significantly change the microstructural images of this zone, which is why it is hard to generalize, as did the authors of papers [1, 2, 11]. Apart from releasing some ingredients (CSH gel, polymer fibres, portlandite) and their concentration on the surface of a bar, it is possible to observe either local formation of hydrates which are a platform between steel and concrete, or thin layers tightly adjoining steel and successive parallel layers, overlapping or mutually penetrating within the 30 – 50 μm zone from the reinforcement surface.

In the course of conducting own research, it was observed that during the “pull-on” test, there were two phases of contact operation, i.e., phase of breaking the bonds of chemical nature (linear chart, stresses proportional to force) and the friction phase (charts disproportionate relative to force or almost horizontal, or at an angle just smaller than the one recorded in the first phase). A low value of this angle

indicates the exhaustion of the contact load bearing capacity in the first phase (1.8% SP + 10% SF is of such nature – matrix durable but fragile; minor force causes definite bar protrusion), high value with a contact load bearing capacity unexhausted in the first phase (flexible matrix, e.g., with an addition of polymer emulsion).

The matrix content, structure of the interface, as well as its mechanical features directly impact the durability of a reinforced concrete structure.

The experimentally obtained tangential stress values given in Table 3 are average values. Limit values, obtained through statistical estimation, assumed as a 5% quantile under an assumption of normal probability distribution of estimated values are used as computational values in engineering practice [12]. Shear values depend only on the concrete class and are, respectively:

C20/25 – 1.4 MPa, C30/37 – 2.2 MPa, C35/45 - 2.6 MPa, C50/60 - 3.0 MPa.

As results from Table 3, the actual values are much higher and for concrete classes C30/37 to C35/45, taking into account the safety coefficient 3 are: standard mortar – 4.78 MPa, modification with SP + SF – 6.37 MPa, mortar with w/c = 1 – 1.59 MPa, mortar with polymer emulsion – 4.78 MPa, mortar with NITCALTM - 6.37 MPa.

If we were to take actual adhesive force values into account in the engineering process, then the currently calculated, the so-called bar anchoring lengths would be significantly shortened, which would result in a more rational design of the structure.

Acknowledgements

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