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Interfacial transition zone in cementitious materials

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Abstract

Subjected to interfacial transition zone (ITZ) examinations were concretes made from gravel and dolomite aggregates in a natural state, and concretes made from the same aggregates coated with paraffin, and also concretes made from the same aggregates with a silica fume admixture. In the case of the gravel concrete, silica fume was added only on the aggregate side, i.e., by taking off 10% of sand in relation to the cement mass and substituting it with silica fume. In the case of the dolomite concrete, silica fume was added on the aggregate side, and also on the cement side. Fracture toughness tests were carried out according to Mode I (tension at bending) following the RILEM Draft Recommendations [Determination of fracture parameters (K_{Ic}^S and $CTOD_C$) of plain concrete using three-point bend tests, RILEM Draft Recommendations, TC 89—FMT, Fracture Mechanics of Concrete—Test Methods, Materials and Structures, 23 (1990) 457–460]. The critical values of stress intensity factors, K_{Ic}^S, and the critical values of crack tip opening displacement, $CTOD_c$, have been determined. The paraffination of coarse aggregate grains, eliminating the adhesion at the aggregate–cement paste interface, caused significant drops in the values of critical stress intensity factor, K_{Ic}^S, as well as those of the critical crack tip opening displacement, $CTOD_c$. This indicates a predominant effect of that layer on the mechanical properties of concretes, and also a high sensitivity of the fracture mechanics parameters to changes within the structure of the aggregate–cement paste transition zone.

Keywords: Interfacial transition zone; Fracture toughness; Silica fume; Concrete

1. Introduction

In cement-based materials, microstructural defects and discontinuities occur, which cause stress concentrations under load in various locations in the volume of these materials. The mechanical parameters of concrete are influenced by the properties of individual constituents, their contents, as well as their interaction and spatial configuration.

In ordinary concrete, aggregate is the least deformed, therefore it is here that stresses are concentrated which are then transferred to the aggregate–cement paste interfacial transition zone (ITZ). According to numerous researchers, the thickness of the ITZ is 40–50 μm. The adhesion between aggregate and cement paste within the transition zone is a factor that governs the concrete strength [1].

The contemporary knowledge in the subject of the ITZ is presented in numerous articles, including three collective studies [2–4], the publication of RILEM TC 108 Technical Committee [5], and in transactions of the RILEM conference [6]. The resources of this knowledge and the intensity of studies conducted do not mean, however, an elimination of doubts and disagreements in the views of different authors on the effect of ITZ on the properties of concretes.

It appears from the studies carried out that the ITZ differs variations in structure from the cement paste that occurs at a greater distance from the aggregate [7–10]. The studies have found that the ITZ has a complex structure; large flat $Ca(OH)_2$ crystals form within this structure, perpendicular to the surface of aggregate grains, which results in the formation of a highly porous structure in the cement paste. This favors the formation of microcracks within this layer and the propagation of microcracks under load.

The forces of adhesion of coarse aggregate grains to cement paste are commonly believed to have a twofold nature. On one hand, these are physical forces whose magnitude depends on the topography of aggregate grain surface and on the grain shape [11], and on the other hand, they are the forces of chemical bonds created at the aggregate–cement paste interface. The latter occur when the aggregate is chemically active towards the cement paste [12,13].

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Table 1
Masses of the constituents in 1 m^3 of concrete mixtures, in kilograms (kg)

Concrete type	Cement	Water	Aggregate, paraffinated aggregate	Silica fume	Superplasticizer
G, G_p	321.6	139.5	2002.7	–	–
GM, GM_p	321.6	139.5	1970.5	32.2	10.6
D, D_p	338.2	159.2	1989.4	–	–
DM, DM_p	338.2	159.2	1955.6	33.8	11.2
DMU, DMU_p	304.4	159.2	1989.4	33.8	–

The composition and structure of the transition zone can be intentionally modified to enhance the strength of the aggregate–cement paste interface. Studies were conducted, which included the preconditioning of aggregate grains by coating the grains with a water–glass and $CaCl_2$ mixture, increasing grain roughness, or increasing the cement paste density by introducing silica gases [14]. Improvement in the properties of the transition zone can also be achieved by introducing silica fume to the concrete mixture, which enhances the adhesion of the paste to aggregate grains. In addition to its pozzolanic properties, silica fume also fills micropores present in the cement paste, thereby improving the mechanical properties of the paste within the transition zone and enhancing the quality of the aggregate–cement paste interface [15].

2. Scope of investigations

The paper presents the results of investigations into the effect of the aggregate–cement paste transition zone on the fracture mechanics parameters as determined according to Mode I following the guidelines of the RILEM Draft Recommendations [16].

Subjected to examinations were two types of concrete from: gravel aggregate and (chemically active) dolomite aggregate. From each of the aggregates, basic series of concrete mixtures (with a thick-plastic consistency) were prepared, as well as series in which the surfaces of coarse aggregate grains had been coated with paraffin. Concretes with an admixture of silica fume were also examined.

In the case of dolomite and gravel concretes, series were prepared in which silica fume dosed as 10% cement replacement or as 10% addition to cement-fix throughout.

For the preparation of all concrete mixtures, the CENII/A-S 32.5R slag Portland cement and aggregate with a grain size of 16 mm and a sand point of 33% were used.

Table 2
Compressive strength of concretes

Gravel concrete	G	GM	G_p	GM_p	
R (MPa)	48.2	61.6	23.3	29.8	
Dolomite concrete	**D**	**DM**	**DMU**	**D_p**	**DM_p**	**DMU_p**
R (MPa)	53.0	65.3	55.3	23.5	27.1	24.6

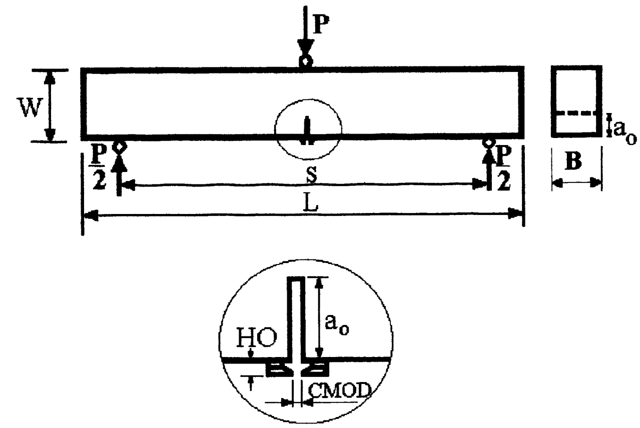

Fig. 1. A schematic drawing of specimen for tests according to Mode I, HO — caliper gauge holder thickness, CMOD — crack opening displacement.

Masses of the components in 1 m^3 of concrete mixtures are provided in Table 1.

2.1. Designations of concrete mixtures made from natural-state aggregates

G — gravel concrete, GM — gravel concrete with a silica fume admixture on the aggregate side (substituting 10% of the cement mass with silica fume), D — dolomite concrete, DM — dolomite concrete with a silica fume admixture on the aggregate side, DMU — dolomite concrete with silica fume on the cement side (substituting 10% of the cement mass with silica fume).

The remaining concrete mixtures from paraffinated aggregates had an identical composition to that in the case of natural-state aggregates.

2.2. Designations of concrete mixtures made from paraffinated aggregates

G_p — gravel concrete, GM_p — gravel concrete with silica fume on the aggregate side, D_p — dolomite concrete, DM_p — dolomite concrete with silica fume on the aggregate

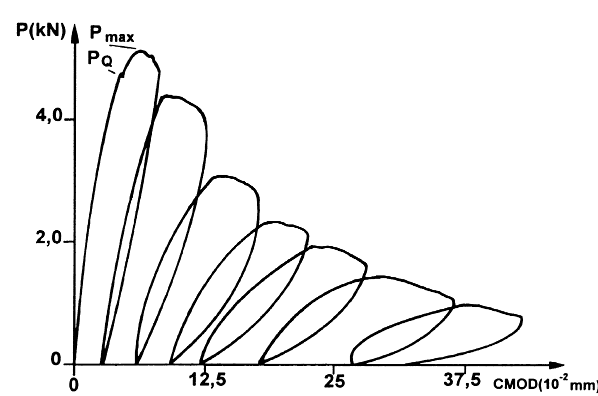

Fig. 2. An example diagram of CMOD vs. load.

Table 3
Fracture mechanics parameters

	Gravel concrete					
	Natural-state aggregate		Paraffinated aggregate			
Fracture mechanics parameters	G	GM	G_p	GM_p		
$K_{Ic}^S \pm s$ (MN $m^{-3/2}$)	2.44 ± 0.16	3.33 ± 0.22	1.24 ± 0.03	1.46 ± 0.12		
$CTOD_c \pm s \cdot 10^3$ (cm)	3.53 ± 0.35	3.85 ± 0.34	2.39 ± 0.34	3.11 ± 0.41		
	Dolomite concrete					
	Natural-state aggregate			Paraffinated aggregate		
Fracture mechanics parameters	D	DM	DMU	D_p	DM_p	DMU_p
$K_{Ic}^S \pm s$ (MN $m^{-3/2}$)	2.53 ± 0.17	3.50 ± 0.24	3.09 ± 0.43	1.48 ± 0.24	2.02 ± 0.22	1.64 ± 0.22
$CTOD_c \pm s \cdot 10^3$ (cm)	2.43 ± 0.15	3.35 ± 0.17	3.11 ± 0.40	2.22 ± 0.28	2.78 ± 0.28	2.32 ± 0.30

side, DMU_p—dolomite concrete with silica fume on the cement side.

In the series with silica fume added on the aggregate side (GM, DM), because of worsened mixing capability, the plasticizer was added in the amount 3.3% in relation to the cement mass. The plasticizer was not added in the paraffinated aggregate series with silica fume (GM_p, DM_p).

Test samples were demoulded after 24 h; then, they were kept in laboratory conditions for 27 days. From each concrete mixture, five specimens were made to be tested for compressive strength, and seven specimens to be tested according to Mode I of fracture.

Compressive test results are summarized in Table 2.

3. Fracture toughness tests

Tests according to Mode I of fracture were carried out using test specimens with dimensions as given in the RILEM Draft Recommendations [16]: W = 150 mm, B = 80 mm, L = 700 mm, S = 600 mm, a_0 = 50 mm (Fig. 1).

An MTS 810 hydraulic testing machine was used in the tests. Loading rate was selected so that the maximum load was reached in approximately 5 min. The applied load was reduced at approximately 95% post-peak load. After reducing the load to zero, the test specimen was loaded again. For each test specimen, six to eight loading–unloading cycles were completed while recording plots of the loading force as a function of crack mouth opening displacement (CMOD). An example plot of CMOD vs. load increment is shown in Fig. 2. Based on the plots obtained for each test specimen, the following quantities were determined: Young's modulus, E; critical effective crack length, a_c; critical stress intensity factor, K_{Ic}^S; and critical crack tip opening displacement, $CTOD_C$.

Critical stress intensity factor, K_{Ic}^S, was calculated from the following relationship Eq. (1):

$$K_{Ic}^S = 3(P_{max} + 0.5w)\frac{S(\Pi a_c)^{1/2}F(\alpha)}{2WB}, \qquad (1)$$

in which [Eq. (2)]:

$$F(\alpha) = \frac{1.99 - \alpha(1-\alpha)(2.15 - 3.93\alpha + 2.7\alpha^2)}{\sqrt{\Pi^{1/2}(1+2\alpha)(1-\alpha)^{3/2}}}, \qquad (2)$$

where: P_{max} = maximum load, $\alpha = a_c/W$, $w = W_oS/L$, W_o = specimen weight [N], S, a_0, W, B, L—according to Fig. 1.

Critical crack opening displacement $CTOD_c$ was determined from the relationship below Eq. (3):

$$CTOD_c = \frac{6P_{max}Sa_cV_1(\alpha)}{EW^2B} \times [(1-\beta)^2 + (1.081 - 1.149\alpha)(\beta - \beta^2)]^{1/2}, \qquad (3)$$

where: $\beta = a_0/a$ ($a = a_0$ before loading).

The results of tests according to Mode I of fracture are summarized in Table 3.

4. Discussion

The investigations showed that the paraffination of coarse aggregate caused a drop in all values of investigated parameters.

In the case of critical stress intensity factor, K_{Ic}^S, for gravel concretes:

- Without silica fume—from 2.44 to 1.24 MN $m^{-3/2}$ (by 49.2%),
- With silica fume added on the gravel side—from 3.33 to 1.46 M $Nm^{-3/2}$ (by 56.2%).

A similar relation was observed for dolomite concretes. The paraffination of aggregate resulted in the following drops in K_{Ic}^S values in concretes:

- Without silica fume—from 2.53 to 1.48 MN $m^{-3/2}$ (by 41.5%),
- With silica fume added on the aggregate side—from 3.50 to 2.02 MN $m^{-3/2}$ (by 42.3%), and

- With silica fume added on the cement side—from 3.09 to 1.64 MN $m^{-3/2}$ (by 46.9%).

The examinations for critical crack tip opening displacement, $CTOD_c$, revealed drops in $CTOD_c$ values as a result of paraffination for all concretes, as follows:

- For gravel concrete without silica fume—from $3.53 \cdot 10^{-3}$ to $2.39 \cdot 10^{-3}$ cm (by 32.3%),
- For gravel concrete with silica fume added—from $3.85 \cdot 10^{-3}$ to $3.11 \cdot 10^{-3}$ cm (by 19.2%),
- For dolomite concrete without silica fume—from $2.43 \cdot 10^{-3}$ to $2.22 \cdot 10^{-3}$ cm (by 8.6%),
- For dolomite concrete with silica fume added on the aggregate side—from $3.35 \cdot 10^{-3}$ to $2.78 \cdot 10^{-3}$ cm (by 17.0%), and
- For dolomite concrete with silica fume added on the cement side—from $3.11 \cdot 10^{-3}$ to $2.32 \cdot 10^{-3}$ cm (by 25.4%).

The paraffination of coarse aggregate grains caused also considerable drops in compressive strength (by over 50%). These drops for particular series of concretes were as follows:

- For gravel concrete without silica fume—from 48.2 to 23.3 MPa (by 51.6%),
- For gravel concrete with silica fume added—from 61.6 to 29.8 MPa (by 51.7%),
- For dolomite concrete without silica fume—from 53.0 to 23.5 MPa (by 55.7%),
- For dolomite concrete with silica fume added on the aggregate side—from 65.3 to 27.1 MPa (by 55.5%), and
- For dolomite concrete with silica fume added on the cement side—from 55.3 to 24.6 MPa (by 58.5%).

The tests carried out showed that the aggregate–cement paste transition zone had a predominant effect on the mechanical parameters of concretes. The drops in the values of critical stress intensity factor, K_{Ic}^{S}, critical crack tip opening displacement, $CTOD_c$, and compressive strength, R, as a result of aggregate grain paraffination, ranged from approximately 40% to almost 60%.

Lower values of K_{Ic}^{S}, and higher percentage drops of this parameter due to paraffination, as found in the tests of gravel aggregate concretes, result from the low adherence of the cement paste to the smooth gravel grains. Crack propagation occurred in this case with a lower critical force than in concretes made from broken dolomite coarse aggregate.

The examination of the critical crack opening displacement showed that the crack propagation in the case of gravel concrete occurred at higher values of $CTOD_c$ than for dolomite concrete, and the drops in these parameters due to the paraffination of grains were greater for gravel concrete than for dolomite concrete, which indicates higher brittleness of dolomite concrete.

Gravel grains probably underwent a slight rotation within the matrix in the failure process, which induced the quasi-plasticity phenomenon, visible on the diagrams, and crack propagation at higher values of $CTOD_c$. In the case of dolomite concrete, aggregate grains had good adherence to the matrix, which resulted in the more rapid failure of this concrete, with a higher critical force than for gravel concrete.

The obtained examination results for gravel concrete relate to mechanical adherence, whereas for dolomite concrete, they embrace the sum of the mechanical and chemical components of the adherence of grains to the paste.

The elimination of both types of adherence as a result of coating aggregate grains with paraffin, and the obtained values of strength parameters have proven that the parameters under study are influenced considerably more by the surface topography and the shape of aggregate grains than by the affinity of the aggregate material to the cement paste.

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