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A new concrete composites based on quaternary binder systems reinforced nanosilica with enhanced fracture toughness and reduced interfacial microcracks

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Abstract. The use of basic Supplementary cementitious materials (SCMs) such as fly ash (FA) or silica fume (SF) due to their pozzolanic properties can change the fracture parameters and structure of the Interfacial Transition Zone (ITZ) between the aggregates and the paste. The combined use of SCMs in combination with very active fine particles of nanoadditives, such as nanosilica (NS), can also bring clear benefits in this regard. From above reasons this study investigated the effect of combined use FA, SF, and NS on the main fracture characteristics and width of microcracks (*W*c) of concrete composites based on quaternary binder systems. For this purpose, a part of ordinary Portland cement (OPC) was replaced with FA+SF+NS in volume of 3 different percentages. The following composition of the new composites, based on quaternary binder systems, has been assumed: constant SF content, equal to 10%, and NS in the amount of 5%, whereas variable FA content, the amount of which was respectively: 0, 5 and 15%. The main experiment in this research was three-point bending tests that were performed on notched beams. Fracture toughness was determined using critical stress intensity factor K_{Ic}^{S}. In addition, the manuscript contains analyses of *W*c occurring in the ITZ area of concretes reinforced NS. On the basis of the obtained test results it can be concluded that the proposed modification of the binder composition in the analyzed materials clearly leads to: homogenization of the composite structure, increasing their fracture toughness, and limitation of initial internal microcracks in concrete.
Keywords: concrete composites based on quaternary binder systems; fly ash; fracture toughness; interfacial microcrack; nanosilica; silica fume

1. Introduction

One of the most important issues in the field of concrete and reinforced concrete structures is the ability to select the components of the concrete mix, and then carry out the technological process of molding the composite structure, in such a way, that the concrete after the curing period in a solidified form is characterized by the lowest possible number of initial microcraks (Li *et al*. 2021a, c, 2022a, Garg *et al*. 2021, Golewski 2024a, b, 2022a, e, Fakoor *et al*. 2014, 2019, Celik *et al*. 2022, Golewski and Sadowski 2006, 2012, Tayeh *et al*. 2021, Ashok *et al*. 2017, Marsavina *et al*. 2017).

Having regard to the mechanical properties of cementitious materials, it is important to observe structure defects due to the fact that as stress concentrators, they constitute the cause of crack development and bring about material damage (Pacheco-Torgal 2017, Golewski 2018 2023a, 2022b, Golewski and Gil 2021, Golewski and Szostak 2022, Lata and Kaur 2019a, b). According to literature data, the area of first microcracks initiation in ordinary concretes is the Interfacial Transition Zone (ITZ) between the largest grains of coarse aggregate and the paste (Golewski 2015, Gil and Golewski 2018a, b, Haeri 2015, Haeri and Sarfarazi 2016, Zhang *et al*. 2019). The role of this concrete phase is significant enough, so that according to (Kaloop *et al*. 2022, Zeyad *et al*. 2019, Zhang *et al*. 2020, Oraka and Sayedi 2021, Golewski 2023a b) the mechanical parameters and the material fracture toughness is not only associated with the strength of components which form the concrete structure, but to a large extent, with the parameters of all contact zones in the composite, as well as material defects occurring even before application of the load. Width of microcracks (*W*c) in the ITZ has also a decisive impact on the fracture toughness, as well as degradation processes of structures made of concrete (Li *et al*. 2021b, 2022b, Abu Al-Rub 2012). For these reasons, knowledge concerning the size of material microcracks is important from a scientific, engineering and economic point of view (Golewski 2024c, d, e, 2019a, b, Kurtinaitiene *et al*. 2016, Szostak and Golewski 2018, UzzalHossain *et al*. 2018, Sokhandani *et al*. 2022, Xi *et al*. 2024a, b, Wang *et al*. 2023).

It should be noted that, one of the methods for the design of composite structures with damage constraints is modifying the composition of the concrete mix in such a way to obtain the smallest possible initial defects of the mature composite structure (Wu and Fang 2022, Yang and Kim 2019, Golewski 2022c, d, Singh *et al*. 2019, Choi and Noh 2000, Peride *et al*. 2009, Craciun *et al*. 2008, Kouchaksarai and Rostamiyan 2023). For this purpose, the components of the concrete mix (mainly the binders) should be selected in such a way, as to achieve the synergy effect of interaction between the individual components used

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(Zhang *et al*. 2016, Sokhandani *et al*. 2022). As a result of this, it is possible to obtain a more homogenous structure of the concrete mix, resulting in a more compact structure of the hardened concrete composite (Guan *et al*. 2022, Golewski 2024c, d, e, Golewski and Sadowski 2016a, b, Szostak and Golewski 2023a, b, c, d, e, f, g, 2020, 2021).

Therefore, concretes containing binders with a modified composition have been used for many years. Such modifications consist in replacing part of ordinary Portland cement (OPC) with other mineral materials (Aydogdu 2014, Golewski 2021a b 2017c d 2019c d, Madenci 2021, Fakoor and Shahsavar 2021, Mehri Khansari *et al*. 2019, Xie *et al*. 2022, Rezaee *et al*. 2022).

Substitutes for cement binder are most often additives, and in recent years also nanoadditives (Sohu *et al*. 2022, El-Chabib and Ibrahim 2013, Biricik and Sarier 2014). In the simplest solutions, i.e. concretes based on binary binders, OPC is replaced by only one additional component, e.g. fly ash (FA), silica fume (SF) or metakalonit (MK) (Karim *et al*. 2015, Bhagawati *et al*. 2016). The benefits which have been observed from such solutions include limiting the width of initial microcracks in concretes of this type, reduction of porosity of such materials, and improving their mechanical parameters and fracture toughness (Guan *et al*. 2019 2021, Craciun 2008, 2016, Craciun *et al*. 2006, Golewski 2020a, b, c, Sadowski and Golewski 2018a, b, c). In most cases however, such effects resulted from the strengthening the cement matrix structure through the development of additional C-S-H and C-A-S-H phases (Cui *et al*. 2022, Fu *et al*. 2022, Kaur *et al*. 2023a, b, Trivedi *et al*. 2022, Zhu *et al*. 2022).

Previous studies have shown, among other things, that particularly positive effect on the fracture toughness and microstructure of the ITZ area between the coarse aggregate and the paste has modification of the composition of the cement binder with single additives and nanoadditives, such as: siliceous fly ash (Zhang *et al*. 2016), silica fume (Lou *et al*. 2023), metakaolin (Han *et al*. 2021), grund granulated blast furnace slag (Kim *et al*. 2019), rice husk ash (Khater *et al*. 2016), waste glass (Zhang *et al*. 2016), natural pozzolan (Zhang *et al*. 2021c), limestone powder (Zhang *et al*. 2021a), nanosilica (Zhang *et al*. 2021b), carbon nanotube (Alimoradzadeh *et al*. 2022), graphene (Ren *et al*. 2020), reactive powders (Rahim *et al*. 2022), C-S-H seeds (Kaloop *et al*. 2022).

Benefits in reducing the microcracks in the ITZ have been also obtained when replacing mineral aggregates with recycled aggregates (Sun *et al*. 2022, Wan *et al*. 2025), coral aggregates (Zhang *et al*. 2019) or limestone aggregates (Golewski 2025, 2022a, b, c, d, e, Golewski *et al*. 2025). However, more advanced solutions in this field of material engineering rely on the substitution of cementitious binder by a combination of two or even three active Supplementary Cementitious Materials (SCMs) (Yuan *et al*. 2022). Such modifications are referred to as concretes based on ternary or quaternary binders (Basak Dipta *et al*. 2023).

It should also be mentioned that material modification in the form of concretes based on multi-component binders also includes pro-ecological activities. It has been proven that by reducing the consumption of OPC and the possibility of using waste materials as a substitute for the binder, such solutions cause (Ramesh *et al*. 2021):

• significant reduction of CO_2 emissions,
• reduction of electricity and heat Energy,
• the possibility of waste management.

Due to the fact that material modification of concrete by a combination of several SCMs simultaneously contributes to both the improvement of properties of the composites with cement matrix, and the development of sustainable construction, the article presents the results of experiments assessing:

• fracture toughness of new concrete composites based on quaternary binder systems reinforced NS,
• the size of microcracks in the ITZ between the coarse aggregates and the paste in the concretes in question.

Basing on the results of the researches conducted it will be possible to obtain information about the possibility of effective modification of concrete composites in terms of obtaining modern, innovative and ecological materials, with increased fracture toughness and durability and increased reliability of work.

Experiments have been planned for four types of concrete. Three of them were a new concrete composites based on quaternary binder systems, while the fourth was control concrete. Modification of the binder composition in modified concretes has been performed by substituting OPC with the two most commonly used mineral additives, i.e. siliceous fly ash (FA) and non-condensed silica fume (SF). In addition, as an element of modern nano-modification, synthetic nanosilica (NS) was used.

Thee following composition of the new concrete composites has been assumed:

• constant SF content, equal to 10%, and NS in the amount of 5%,
• variable FA content, the amount of which was respectively: 0, 5 and 15%.

Basing on such assumptions, it was possible to determine the impact of basic SCMs, i.e. FA for the fracture toughness and size of microcracks in the ITZ area of the coarse aggregate with the cement matrix in concretes containing silica additives.

2. Experimental procedures

2.1 Materials

In order to prepare new concrete composites based on quaternary binder systems were used:

• Type I OPC (CEM I 32.5R), which met the requirements of standard provision EN 197-1:2011 (2011),
• SCMs such as: FA, SF and NS.

The specific gravity of OPC, FA, SF and NS are 3.11, 2.14, 2.21 and 1.10 g/cm^3 respectively. The Blaine fineness of the OPC, FA, SF and NS are 0.33, 0.35, 1.40 and 200 m^2/g respectively. Fig. 1 shows a photos of a FA, SF and NS replacing the OPC. On the other hand Table 1 provides where all binders used came from.

A local well graded pit sand and a gravel stones, from the Lublin area in Poland, with a maximum nominal

(**a**)

(**b**)

(**c**)

Fig. 1 A view of SCMs used in order to prepare a new concrete composites based on quaternary binder systems: a) FA, b) SF, c) NS.

Table 1 The origin of binder used

Origin	Kind of binder			
	OPC	FA	SF	NS
Country		Poland		South Korea
City	Chełm	Puławy	Łaziska	Seul
Manufacturer	Cement plant	Power station	Ironworks	Chemical company

aggregate size of 2 and 8 mm were used as fine and coarse aggregates, respectively. The relative specific density and bulk density of the coarse aggregate were 2.65 and 2.25 g/cm^3 respectively, whereas the fine aggregate had a relative specific density and bulk density of 2.60 and 2.20 g/cm^3 respectively. On the other hand, the modulus of elasticity for both aggregates were 330 GPa.

A new generation of polycarboxylic based high-range water reducer admixture superplasticizer (SP) – STACHEMENT 2750 was used to enhance concrete workability and stability. Recommended dosage for SP was 1.8 % of cementitious materials.

In order to prepare concrete mixtures tap water was also used, which met the requirements of standard provision EN 1008:2002 (2002).

2.2 Test specimen preparation and mix proportions

The proportions of mixtures for reference concrete and new concrete composites based on quaternary binder systems reinforced NS per volume of one cubic meter presents Table 2. In this study, four series of mix design with constant water to binder ratio of 0.40 were considered. It should be noted that the amount of superplasticizer used as a percentage of the total cementitious materials and the amount of water in the superplasticizer was also considered in the ratio of water to cementitious materials.

For labelling concrete mixes according to Table 1, Mix-1 represents plain concrete without SCMs (reference sample), whereas Mix-2, Mix-3 and Mix-4 indicates new composites based on quaternary binder systems reinforced NS. These concretes containing constant content of 10% SF and 5% NS and variable content of FA in the amount of: 0, 5 and 15% of the OPC volume, respectively.

To manufacture concrete mixtures, first – the aggregates including pit sand and gravel were mixed in a drum mixer for 120 s. Then – the cementitious materials were mixed with the pozzolans used (OPC+FA+SF), and added to the mixture to be mixed in the mixer for the next 180 s. Then – a mixture of SP, part of the water and NS was gradually poured into the mixer and stirred for 120 s. At the end – the remaining water was added and all components have been mixed for 120-180 s to obtain a homogenous mixture.

After final preparation, the fresh mixture is poured into molds and compacted on a vibrating table. The concrete beams with initial cracks are then cast for evaluating the fracture toughness – critical stress intensity factor, K_{Ic}^{S}.

After finishing, the specimens were covered with wet fabric and stored in the casting room at 20 $\pm$ 2°C. In the next step, the specimens were demolded after 48 h and kept in a water tank for the first 14 days. For the next 2 weeks, the specimens were cured under laboratory conditions. Then – they have been tested. Details of specimens preparation for fracture toughness tests and microstructural examination are given in Section 2.3.

2.3 Test procedures

2.3.1 Fracture toughness investigation

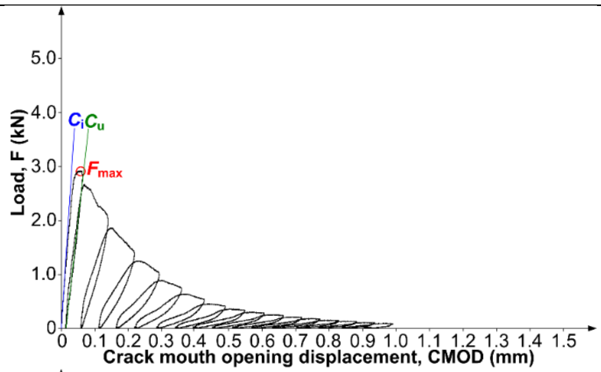
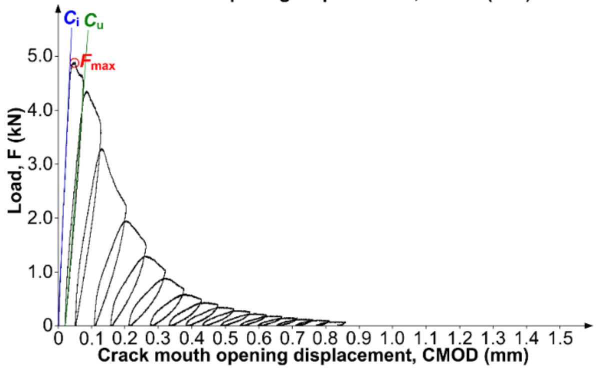
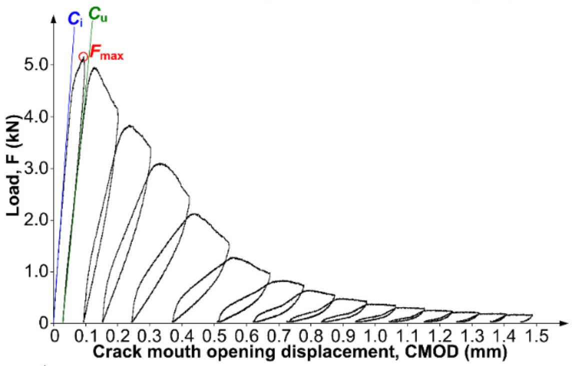
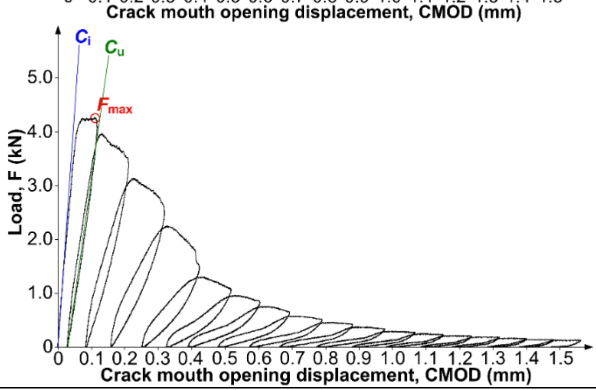
In this research, in order to evaluate the main fracture parameter for concrete composites, i.e. critical stress intensity factor – K_{Ic}^{S}, for each mixture, six notched beams with the same dimensions of 80 × 150 × 700 mm (depth × width × length) with a span of 600 mm were made (Figure 2a). In addition, Figure 2 shows a sample of concrete beams tested for fracture toughness analysis. The initial vertical notch with a constant width of 3 mm was created by placing a steel plate in the middle of the beam in the tensile direction of the specimens.

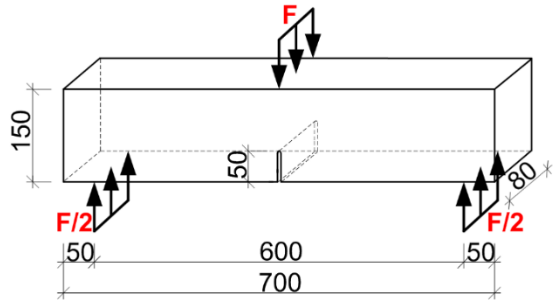
As can be seen from Fig. 2, the loading application was through a concentrated load at the middle of the beam

Table 2 Proportion of concrete mixtures

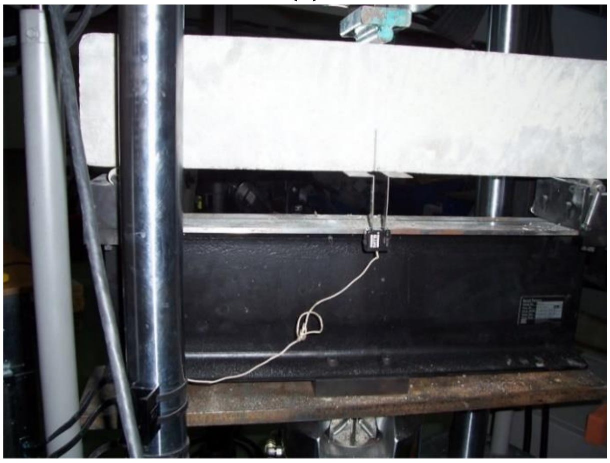
Percentage composition of the mix	Mix label	Weight of constituents of concrete (kg/m^3)							
		OPC	FA	SF	nS	Water	SP	Sand	Gravel
100% OPC	Mix-1	352	0	0	0	141	0	676	1205
85% OPC+0%FA+10%SF+5%nS	Mix-2	299.2	0	35.2	17.6	141	6	676	1205
80% OPC+5%FA+10%SF+5%nS	Mix-3	281.6	17.6	35.2	17.6	141	6	676	1205
70% OPC+15%FA+10%SF+5%nS	Mix-4	246.4	52.8	35.2	17.6	141	6	676	1205

Table 3 Exemplary *F–CMOD* curves with significant details for analysed composite

Mixture label	Typical *F-CMOD* curve
Mix-1	
Mix-2	
Mix-3	
Mix-4	



(**a**)



(**b**)

Fig. 2 Specimen used in the fracture toughness tests: a) static scheme with dimensions and details, b) test setup for $\boldsymbol{K}^{S}_{\mathrm{Ic}}$ determination, dimensions in mm, *F*-Force

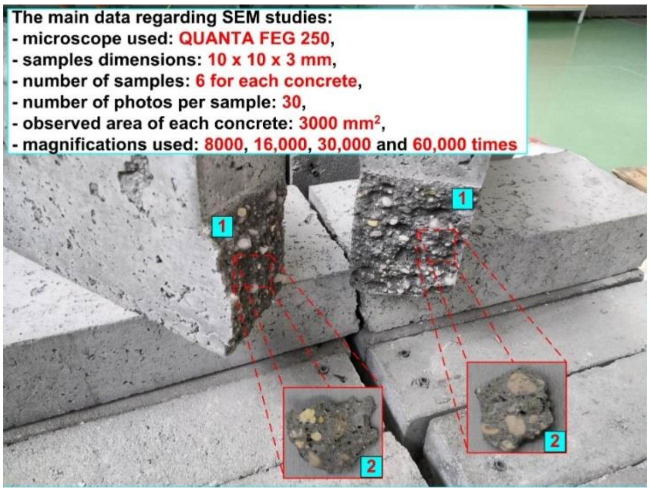


Fig. 3 Sampling scheme for SEM tests including the data relevant in this studies: 1-cracked surface of beam after conducted tests, 2- view of sample for SEM tests

span with a displacement-controlled strategy. Also, the load and deformation of the beam specimen were recorded simultaneously in each time period.

The fracture toughness test was carried out in accordance with the RILEM Draft recommendations TC-89 FMT (1990) using MTS 810 testing machine (Figure 2b). In addition the crack opening sensor that was the MTS clip gage axial extensometer 632,03F-3 was used in order to measure width of the initial crack opening during the tests (Figure 2b). During the tests carried out on the MTS 810 press, Force (*F*) – Crack mouth opening displacement (*CMOD*) curves were recorded (Lou *et al*. 2022, Lu *et al*. 2022).

The analysed fracture toughness K^{S}_{Ic} was determined with the use of obtained diagrams *F*–*CMOD* and the detailed formulas given in RILEM Draft recommendations TC-89 FMT (1990). It should be added that in order to determine the K^{S}_{Ic} parameter, it was necessary to read the following data from the *F-CMOD* charts (Table 3): maximum load obtained in the tests, marked in red ($F_{\max}$), tangent in the first phase on the *F*–*CMOD* relationship, highlighted in blue (*C*i) and tangent in the second phase on the *F*–*CMOD* relationship, highlighted in green (*C*u). They have been marked on the exemplary charts for each of the analyzed composites and summarized in Table 3.

2.3.2 Microstructural studies

In order to trace the combined impact of FA, SF and NS particles on the width of microcracks – *W*c at the ITZ of the coarse aggregate with the cement matrix in new concrete composites based on quaternary binder systems, micro-structural analysis has been carried out (Naija *et al*. 2022). Experiments were performed after 28 days of curing of composites. The impact of the applied SCMs on the analyzed parameters has been assessed using a scanning electron microscope (SEM). Samples for SEM analysis were taken from beams damaged after fracture toughness tests. Sampling scheme for these experiments, including a view of two samples prepared for SEM tests is shown in Figure 3. In addition, Figure 3 contains relevant data related to the samples used, and the main assumptions adopted in the SEM tests.

3. Results and discussion

3.1 Fracture toughness

The exemplary *F*–*CMOD* curves of the concrete beams, prepared on binders including SCMs, under three-point flexural loads are shown in Table 3. Important data such as: $F_{\max}$, *C*i and *C*u are also shown in each of the exemplary charts.

On the basis of the compiled graphs in Table 3, it can be concluded that the samples in all concretes were damaged at the $F_{\max}$ force, which were:

• about 3.0 kN in the case of unmodified concrete, i.e. Mix-1,

• almost 5.0 kN in the case of replacing part of the basic binder with silica additives only, i.e. SF+NS – for Mix-2,

• from 4.0 to over 5.0 kN when using a combination of three active SCMs simultaneously, i.e. SF+FA+NS – for Mix-3 and Mix-4.

In addition, by analysing the slope of the *F*–*CMOD* curves, assessed on the basis of the shape of the *C*i and *C*u tangents, it can be concluded that the proposed material modification changed the behaviour of the composites in the process of their destruction. Definitely the most slender

Table 4 Results of fracture toughness K_{Ic}^{S}

Mixture label	$\overline{K_{Ic}^{S}}$ (MNm$^{-3/2}$)	(MNm$^{-3/2}$)	(%)	K_{Ic}^{S}, max. (MNm$^{-3/2}$)	K_{Ic}^{S}, min. (MNm$^{-3/2}$)
Mix-1	1.06	0.12	5.55	1.15	0.98
Mix-2	1.50	0.17	7.14	1.67	1.38
Mix-3	1.58	0.20	8.28	1.72	1.41
Mix-4	1.39	0.23	9.15	1.61	1.18

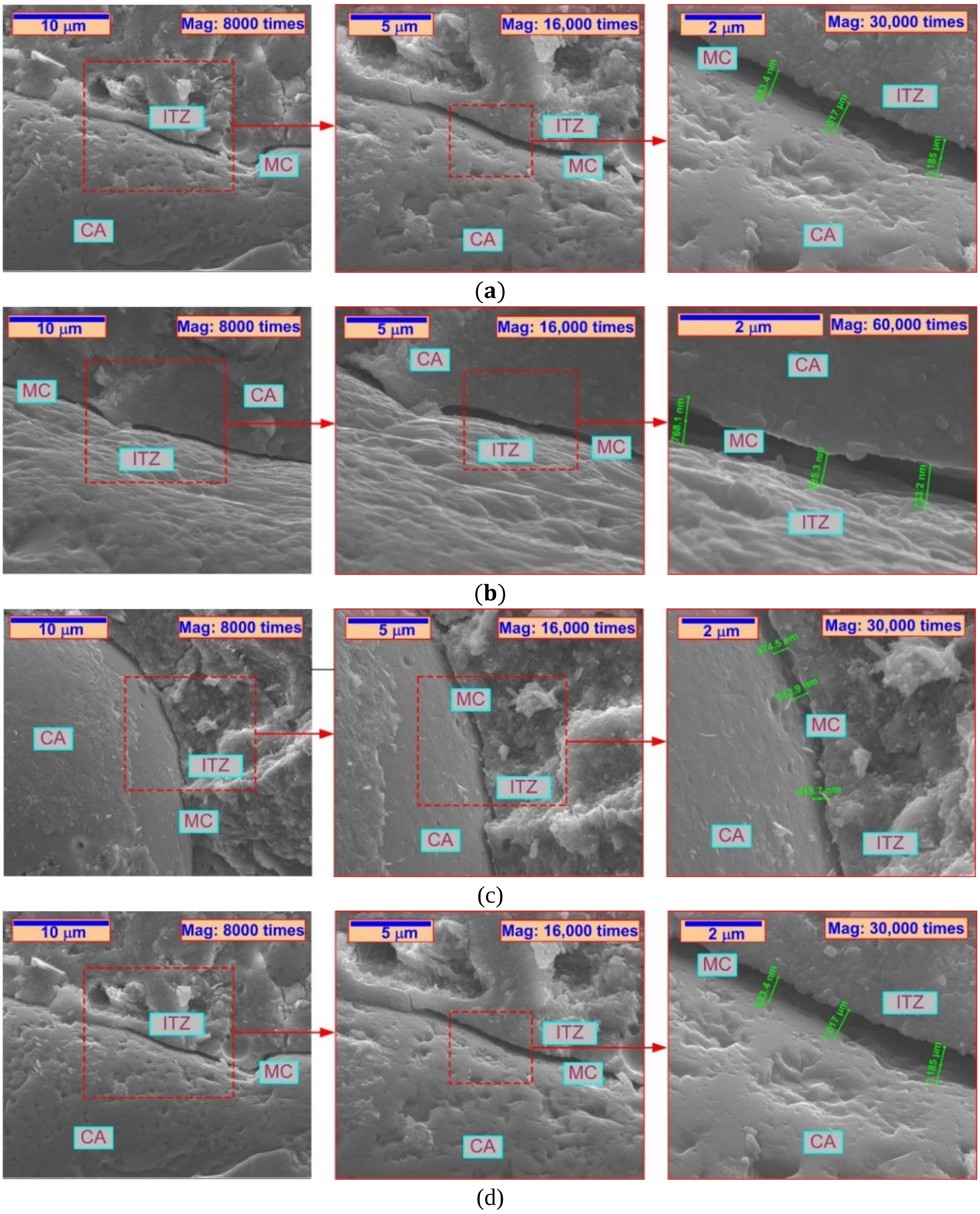


Fig. 4 SEM microimages of analysed composites showing microcracks in the ITZ area: a) Mix-1, b) Mix-2, c) Mix-3, d) Mix-4, CA-Coarse aggregate, MC-Microcrack, P-Pore, E-Ettringite, CH-Calcium Hydroxide

graphs were those obtained for Mix-2 series concrete. In addition, the initial crack development process visible on the load-unload loops was relatively short. In the case of cyclic material damage the effect of its "flow" was not observed. However, such behaviour was clearly visible in the case of composites of the Mix-3 and Mix-4 series (Table

Table 5 Results of width of microcrack *W*c

Mixture label	$\overline{Wc}$ (μm)	δ (μm)	v (%)	*W*c max. (μm)	*W*c min. (μm)
Mix-1	1.13	0.05	4.63	1.19	1.01
Mix-2	0.70	0.08	6.89	0.79	0.62
Mix-3	0.59	0.09	7.32	0.68	0.50
Mix-4	0.79	0.12	8.12	0.93	0.68

3). This proves that the concrete of the Mix-2 series was the most brittle of all the analyzed composites.

The addition of FA to the binder composition caused that composites containing 3 active SCMs had signs of quasi-plastic behaviour during the process of damage and destruction. This was more evident in concrete with higher content of FA, i.e. 15%. A similar effect was also observed in the reference concrete (Table 3). It should be noted that the obtained results also confirm the assessment of the brittleness of the composites in question carried out on the basis of the brittleness index (BI) analysis. In the study (Golewski 2023d) it was shown that concrete containing SF+NS additives was characterized by the highest brittleness, while the remaining three composites showed signs of quasi-plasticity. As a result of the addition of FA to the binder composition, the value of the brittleness index increased, indicating a reduced brittleness of the material (Golewski 2023d). Among the composites based on quaternary binder systems, quasi-plastic behaviour was the most evident in Mix-4 series concrete (Table 3).

The observed average fracture toughness – $\overline{K_{Ic}^{S}}$, are shown in Table 4. Statistical parameters, i.e. standard deviation – δ and coefficient of variation – v, as well as the dispersion of the results, $K_{\mathrm{Ic}}^{\mathrm{S}}$, max. and $K_{\mathrm{Ic}}^{\mathrm{S}}$, min. are also given in Table 4.

Based on the analysis of the results contained in Table 4, it can be concluded that the highest fracture toughness had Mix-3 series concrete – containing SF and NS, as well as FA at the level of 5%. Equally favourable results of $K_{\mathrm{Ic}}^{\mathrm{S}}$ were obtained in the case of Mix-2 series concrete. Larger addition of FA, i.e. in the amount of 15%, resulted in a weakening of the composite structure and decrease of the $K_{\mathrm{Ic}}^{\mathrm{S}}$ value for this material. Nevertheless, fracture toughness of the Mix-4 series concrete was still significantly higher than $K_{\mathrm{Ic}}^{\mathrm{S}}$ for the reference concrete (Table 4).

The obtained results of fracture toughness prove the obvious benefits of the proposed material modification. This action caused an increase of the $K_{\mathrm{Ic}}^{\mathrm{S}}$ parameter in cement concretes containing combined SCMs, from 30 to almost 50% (Table 4).

In addition, it should be stated that with a more advanced modification of the basic composition of the binder, the obtained $K_{\mathrm{Ic}}^{\mathrm{S}}$ results were less convergent, i.e. standard deviations and coefficient of variations of the analyzed parameter for composites based on quaternary binder systems clearly increased (Table 4).

3.2 Interfacial microcracks

Fig. 4 shows exemplary, representative images of the microstructure of individual tested composites. In accordance with the assumptions described in the first section of the manuscript, the analysis focused on measurements of the width of microcracks (*W*c) in the ITZ area of each concrete. Each of the photos shows characteristic areas at two basic magnifications, i.e. 8000 × and 16,000 ×. In addition, in the case of concretes based on quaternary binder systems reinforced NS, the places of ITZ between the coarse aggregate and the paste were imaged at additional – very large magnifications applied, i.e. 30,000 × or 60,000 ×. In addition, in order to better highlight the changes in the structure of the analyzed concretes, all significant details observed were also marked on the selected representative photos. These included:

- main phases occurring in the cement matrix,
- places of microcracks in the ITZ area, with the indication of their exact dimensions in three places.

The observed average $\overline{Wc}$ with statistical parameters δ, v and the dispersion of the results *W*c min. and *W*c max. are presented in Table 5.

When analyzing the average *W*c values it should be stated that the modification of the basic structure of the cement matrix present in the reference concrete, Mix-1 resulted in a significant reduction of the analyzed parameter in all composites based on quaternary binder systems (Table 5).

On the basis of the above results, it can therefore be concluded that the proposed modification of the binder composition in the analyzed materials clearly leads to homogenization of the composite structure and limitation of initial internal microcracks in concrete composites. In addition, similarly as in the case of K_{Ic}^{S} value assessment, also in the case of statistical assessment of *W*c, there was a noticeably higher dispersion of results in case of composites based on quaternary binders system reinforced NS (Table 5). However, due to much higher number of results included in the statistical analysis, i.e. 30, the convergence of *W*c value was higher, compared to the results obtained for K_{Ic}^{S} (Table 4). Nevertheless, in both of the analyzed parameters variability of the results did not exceed 10%. Therefore, it can be concluded that their representativeness was at a satisfactory level.

4. An in-depth analysis of the obtained test results

Due to the fact that many researchers have suggested relationships between fracture toughness of concrete composites and the size of structural microcracks, mainly in the ITZ area between the coarse aggregate and the paste (Ferro *et al*. 2015, Akbari *et al*. 2022), in the present study

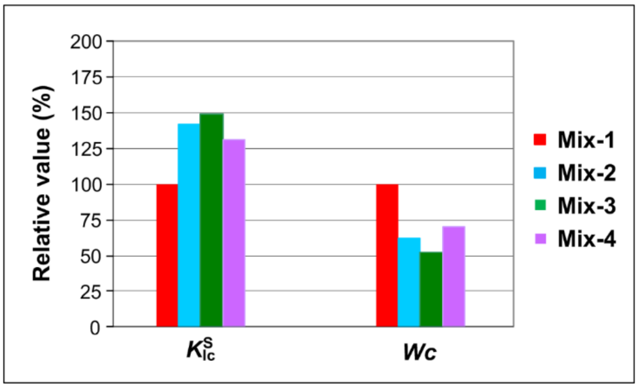


Fig. 5 Relative values of analysed parameters for all composites

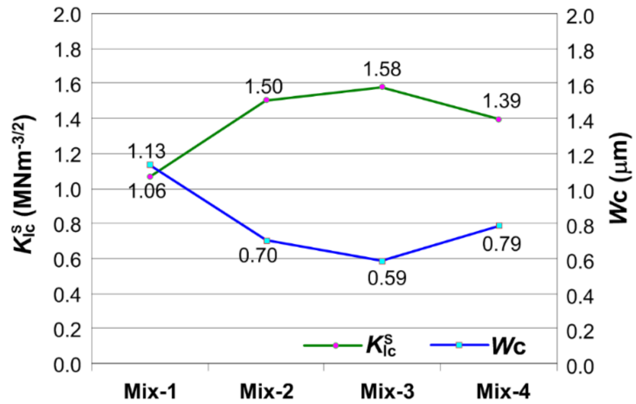


Fig. 6 Changes of analysed parameters as a result of replacing OPC by SCMs

clear relationship between these two factors was presented.

On Fig. 5 it can be seen that addition of SCMs in concrete composites increases the amount of fracture toughness K_{Ic}^S in these materials, while the value of microcrack width *W*c in this case decreases. An increase of the critical stress intensity factor in concretes series from Mix-2 to Mix-4 was observed, by 42%, 49% and 31%, respectively. However, microstructural tests have shown that the size of microcracks in the ITZ areas has decreased in concretes based on quaternary binder systems reinforced NS, appropriately by: 38%, 48% and 30% for the Mix-2 to Mix-4 series of concretes (Fig. 5).

Therefore, a summary analysis of the obtained test results indicates, that there is an inverse relationship between the fracture toughness of the concretes in question, and the level of defectiveness of their structure. The calculated values of the critical stress intensity factor K_{Ic}^S are clearly inversely proportional to the measured *W*c values in the ITZ area between the coarse aggregate and the paste. This interdependence is shown in Fig. 6.

This figure clearly shows that with the change in the composition of the binder in individual composites, both the material fracture toughness and the size of microcracks in the ITZ area change significantly. The K_{Ic}^S parameter increases compared to the values obtained for the Mix-1 series concrete, while the *W*c index is reduced. However, the intensity of changes in both analyzed parameters is closely related to the composition of the binder for a given type of composite. Existing differences may be explained on the basis of microstructural observations.

Based on the observations of the matrices of individual composites, presented in Figure 4, it was found that the control concrete, i.e. Mix-1 series was characterized by the most heterogeneous structure. Most of the phases present in this material were still in the process of reacting. Pores in the cement matrix were also observed (Fig. 4a).

Substitution of part of the cement binder with active pozzolanic additives resulted in a more compact and homogenous structure of the cement matrix in each of the new concrete composites based on quaternary binders reinforced NS. However, the addition of only SF and NS in the concrete of the Mix-2 series caused only a slight change in the morphology of the matrix. Nevertheless, the surface of fracture in this concrete was already clearly more compact than in the concrete of the Mix-1 series (Fig. 4b).

In the concrete of the Mix-3 series, which contained both silica and FA additives in the amount of 5%, the structure of the matrix was clearly compact. In this composite it was possible to observe clearly the synergy effect occurring between the three SCMs used (Fig. 4c).

Increasing the amount of FA in the binder composition resulted in the effect of weakening the quality of the matrix structure. The structure of the cement matrix in the concrete of the Mix-4 series was also less compact than in case of concrete with a lower FA content, i.e. Mix-3.

Therefore, the results of microstructural tests of concrete containing FA confirm the earlier results given, among others by Zhang (1995). On their basis, it can be concluded that the beneficial effect of FA additive in strengthening the structure of the cement matrix becomes evident in case of using this useful waste only to a certain level. In the case of concretes modified by FA only, this is the range not exceeding 20% (Ho *et al*. 1985, Fraay *et al*. 1989, Lam *et al*. 1998). However, in the case of composites based on ternary or quaternary binder systems, the level of OPC substitution by FA should be much lower, preferably at the level of several percent (El-Chabib *et al*. 2013, Patel *et al*. 2016).

In addition, it should be stated that with a more advanced modification of the basic composition of the binder, the obtained K_{Ic}^S and *W*c results were less convergent. This was the most clearly visible in concretes containing all three SCMs (Tables 4, 5). This phenomenon is probably caused by the heterogeneous reaction of FA grains or their partial separation from the matrix structure in the Mix-4 series concrete. This, in turn, has an impact on increased dispersions in the results obtained in concrete with these additives.

On the basis of the above results, it can therefore be concluded that the proposed modification of the binder composition in the analyzed materials clearly leads to:

• homogenization of the composite structure,

• increasing the fracture toughness of materials containing SCMs,

• limitation of initial internal microcracks in concrete composites based on quaternary binder systems reinforced NS.

5. Conclusions

From the above data, it can be stated that the introduction of pozzolanic additives in the form of FA and SF in combination with very reactive nanoadditive, which is NS, accelerate the hardening of concrete composites after 28 days of their curing. At the same time, the most noticeable effect of increasing the fracture toughness, as well as reducing microcracks in the ITZ area in concretes based on quaternary binder systems of concrete series Mix-3 were observed. Therefore, based on the laboratory work performed in this research, it can be concluded that:

• Concretes based on quaternary binder systems reinforced NS are characterized by better fracture toughness K_{Ic}^{S} in comparison to control concrete.

• Addition of three pozzolanic materials, i.e. FA, SF and NS in combination modifies the microstructure in the ITZ area of concrete composites.

• Reference concrete is characterized by greater microcrack width *W*c in the ITZ area in comparison to the concretes based on quaternary blended cements including NS.

• The highest fracture toughness and the smallest microcrack occur in concrete based on quaternary binder systems with the 10%SF+5%FA+5%nS.

• In the case of concretes modified with three active pozzolanic materials, the sizes of microcracks in the ITZ rea between the coarse aggregate and the paste are clearly reduced.

• The values of the critical stress intensity factor K_{Ic}^{S} are clearly inversely proportional to the measured *W*c values in the ITZ area between the coarse aggregate and the paste.

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