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Fracture Toughness of Concrete Containing Fly Ash

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Abstract

The chapter presents results of tests on the effect of the addition of siliceous fly ashes (FA) in the amount of 0, 20, and 30% by weight of cement on the fracture processes in structural concretes. In the course of experiments, measurements of compressive strength of concrete were done as well as fracture toughness for I, II, and III model of cracking was evaluated. During the tests, the effect of age of concretes modified with the additive of fly ashes on analyzed parameters was determined. The experiments were carried out after 3, 7, 28, 90, 180, and 365 days of curing. Fracture toughness of concretes was determined based on the critical stress intensity factors, and then a generalized fracture toughness K_c was determined. The properties of composites with the additive of fly ashes depend on the age of the concrete during tests. Twenty percent additive of fly ashes guarantees high fracture toughness in mature concretes, while the additive of fly ashes in the amount of 30% weight of cement has a beneficial effect on the parameters of concrete only after half a year of curing. Both the 20 and 30% additive of fly ashes significantly reduce the fracture toughness at an early age.

Keywords: fly ash, concrete composite, compressive strength, mode I, II, III fracture, critical stress intensity factor, fracture mechanics, fracture toughness, curing time

1. Introduction

1.1. Purpose and scope of the present study

Among the primary components making up the structure of concrete are Ordinary Portland Cement (OPC), sand, coarse aggregate, water and optional mineral additives, and chemical admixtures. In the last several dozen years, with the development of a new generation of concrete, there has been a significant increase in the production of concrete mixtures containing additives.

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A significant increase in the production of concretes with additions is closely connected with the fact that acquisition of natural raw materials and fuels for the economy in the world is becoming an increasing problem. Therefore, a very important issue is to find cheap and fully useful materials of mineral origin that will be able to replace the most expensive component in the composition of cement, which is the Ordinary Portland Cement (OPC). Furthermore, Ordinary Portland Cement is not considered an environmentally friendly material and consumes natural raw materials such as limestone and natural sand. It should be added that the production of Ordinary Portland Cement not only uses a considerable amount of energy, but also emits a substantial amount of carbon dioxide (CO_2) and other greenhouse gases [1]. The production of one ton of Ordinary Portland Cement requires 4 GJ of energy and emits approximately 1.35 billion tons of CO_2 into the atmosphere annually [2]. It is estimated that the CO_2 released during cement clinkering is around 0.7–0.9 tons per ton of Ordinary Portland Cement, meaning that the cement industry generates around 7% of total CO_2 emissions worldwide [3]. For that reason, research on alternative materials has increased. Nowadays, different types of waste are used as additives to concrete in order to improve its durability, strength, and fracture toughness [4]. Using waste materials in concrete has become a necessity to provide a sustainable environment. Composites using waste so-called green concretes [5] or Eco-friendly concretes [4]. Structures made of green concrete are environmentally sustainable and are constructed in such a way that the total impact on the environment during their full life cycle, including service life, is reduced to a minimum.

One of waste materials is fly ash (FA). As a substitute for Ordinary Portland Cement, many types of fly ash are used [6]. However, the most commonly used fly ash is siliceous fly ash, which is a by-product obtained in the process of hard coal combustion performed in electric power stations and in thermal-electric power stations [7]. During combustion processes, siliceous fly ash is removed by mechanical collectors or electrostatic precipitators as a fine particulate residue from the combustion gases before they are discharged to the atmosphere [8].

Nowadays, fly ash has become one of most promising alternative binders to Ordinary Portland Cement. The fly ash from coal combustion processes is a waste very attractive for recycling because concretes with these additives characterized by, higher i.e.:

- corrosion resistance,
- resistance to high temperatures,
- abrasion erosion resistance,

and lower i.e.:

- shrinkage,
- heat of hydration
- water permeability

The siliceous fly ash has been utilized for many years in cement and concrete production. Namely, in the manufacturing process of concretes, it is possible to replace part of the cement binder in the concrete with fly ash treated as pozzolanically active materials. Advantageous impact of fly ash addition is related to the morphology of its particles (shape and surface properties), pozzolanic activity, and microfiller effect (aggregate void filling).

From above reasons, fly ash utilizations are 39% in the USA and 47% in Europe, whereas the global average is estimated to be close to 25%, which is approximately 200 million tons per annum. Up to date, more than 750 million tons of the fly ash are generated each year in the world [9]. The main areas of development of siliceous fly ash are construction, mining, and terrain management.

The analyses of use of this material in the construction industry reveal that more than 50% of siliceous fly ash is used in the cement industry. Therefore, the fly ash is a subject of intensive research. The first paper, which described the properties of concrete containing the fly ash, was published already in (1937) [10], whereas the first critical review of using fly ash in concrete was published in 1980 [11].

Nowadays, good-quality siliceous fly ash is used for production of Portland-composite cement and plain concretes as well as self-compacting concretes, roller-compacted concretes, or steel-fiber reinforced concretes. Moreover, with the right quality (low loss of ignition (LOI) and high fineness) and quantity (up to 15%) of the fly ash, selection is also possible to use them for the production of polymer concrete and concretes with higher strengths, that is, high-performance concretes and ultra-high-performance concretes.

What effect does the addition of siliceous fly ash have on the basic properties of the concrete mixture and concrete, and how it should be used are described in detail in several monographs [12–14] and numerous scientific publications. Nevertheless, certain features of concrete composites with fly ash have so far been investigated and analyzed very rarely. For example, up till now the effect of fly ash influence on the fracture toughness of the concrete was not investigated systematically. Such studies were carried out rather occasionally.

Therefore, this chapter presents results of macroscopic tests on the effect of the addition of siliceous fly ashes in the amount of 0, 20, and 30% by weight of cement on the fracture processes in structural concretes. In the course of experiments, measurements of compressive strength were done as well as fracture toughness for I, II, and III model of cracking was evaluated. Fracture toughness of concretes was determined based on the critical stress intensity factors for all mode fracture, and then a generalized fracture toughness K_c was determined. During the tests, the effect of age of concretes modified with the additive of siliceous fly ashes on analyzed mechanical parameters was determined. The experiments were carried out after 3, 7, 28, 90, 180, and 365 days of curing.

1.2. Discussion of topic selection and scientific objectives

The properties of concrete materials, including their durability, mainly depend on the structural factors and the interaction between micro- and macro-structure of the material. Cracks and losses are the two basic defects of the concrete structure, which may reduce the load-bearing capacity, leak tight integrity, and stiffness of elements and structures, which in extreme cases may lead to failure and even building catastrophes. It is therefore important for the engineer designing the concrete mix to be familiar with the processes of damage and crack development in structural materials, especially those that are characterized by high brittleness. It allows improvement of the quality of concrete, from which the structures are made, estimation of defects, and determination of their causes. This leads to obtaining composites with the highest durability and reliability.

The overall objective of the conducted scientific analyses was a description of fracture processes in concretes with the siliceous fly ash additive. Concretes with these additives have been used in the industry for approximately 80 years, and in the past two decades, their use in the composites with cement matrix has significantly increased. Therefore, it is obvious that in the course of such a long period of time, it has managed to explore the effect on the basic physical and mechanical characteristics of the siliceous fly ash additive. However, based on literature research, it is clear that in this area of science, there is a clear gap. It is linked to the lack of complete data relating to the analysis of initiation and propagation of microcracks and cracks, in this type of materials, developing during the impact of complex states of stress on concrete.

Description of gradual degradation of brittle materials like concretes is a multi-stage process depending on an internal structure of composite and a level of external loads. A detailed description of particular stages of development of cracks in concrete structure is shown in Section 2. The reduction of strength of concrete results from initial structural defects existing inside material [15]. Additionally, a crucial role on the fracture behavior of concrete plays an aggregate structure [16].

Cracks initiation and propagation in the concrete require the knowledge of all fracture mechanics parameters for modes I, II, and III, that is, K_{Ic}, K_{IIc}, and $KIII_c$. This is due to the fact that fracture is an important feature in concrete at all scales [17]. Moreover, the damage of concrete subjected to complex loading involves strong anisotropy due to its highly heterogeneous nature and geometrically anisotropic characteristic of microcracks. Thus, the three-dimensional (3D) fracture process is generally complicated, that is, an experimental estimation of all fracture mechanics parameters is extremely difficult and can be done for three separate fracture modes: I, II, and III. **Figure 1** defines the three modes of loading, that is:

- Mode I, opening or tensile mode,
- Mode II, sliding or in-plane shear (pure shear) mode, and
- Mode III, tearing or anti-plane shear mode.

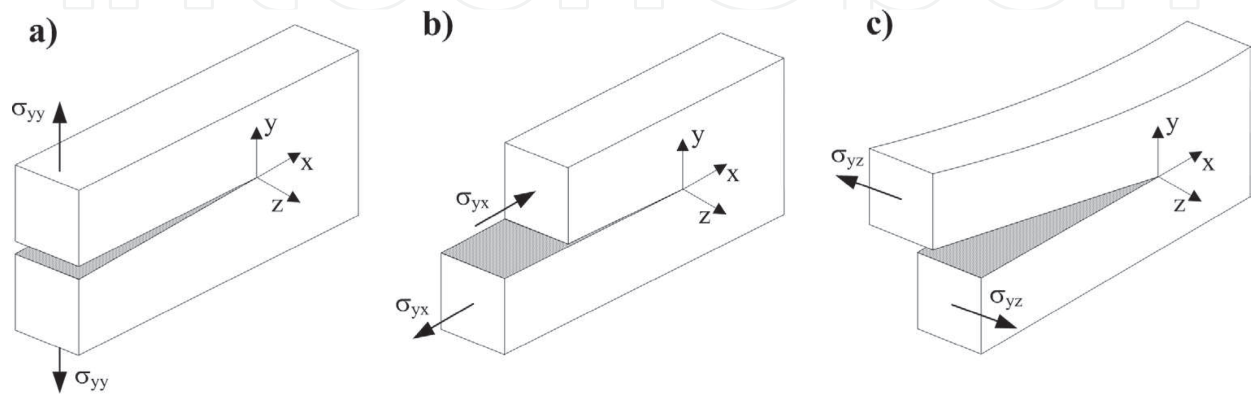

Figure 1. Schemes of the three modes of cracking: (a) mode I, (b) mode II, and (c) mode III.

In general, cracks in real applications are often subjected to a mixed mode loading conditions, whereas fracture may occur under mode I, mode II, mode III, or a combination of them [18]. The result is that in many cases, cracks in structural elements start and develop in the mixed mode I–II loading or I–III loading. A detailed analysis of stress intensity factors for plain concrete is presented by Song et al. [19] for different combinations of pure and mixed modes of cracking to formulate general three-dimensional (3D) fracture criterion. Tests of fracture toughness in complex stress states are associated with the description of the present critical state in the element, which can be written as the following relationship:

$$f\left(K_{Ic}, K_{IIc}, K_{IIIc}\right) = f_c. \tag{1}$$

With the known values of K_{Ic}, K_{IIc}, and K_{IIIc}, it is possible to designate a generalized fracture toughness of the material, K_c, from Eq. (2) [20]. The reason that the stress intensity factor is useful is because fracture (sudden crack growth to failure) occurs when K exceeds a certain critical value, K_c, called exactly the fracture toughness.

$$K_c^2 = K_{Ic}^2 + K_{IIc}^2 + \frac{K_{IIIc}^2}{1-\nu}, \tag{2}$$

where ν is the Poisson's ratio.

Unfortunately, in the assessment of fracture toughness of concretes with fly ash, the available results from experiments relate only to the first mode fracture, that is, tension at bending. Fracture toughness under mode I for plain and high-performance concretes containing siliceous fly ash additives, applying three-point-bending tests, was reported in Refs. [21–23]. No article was found in the literature that would discuss the fracture toughness of concrete with the siliceous fly ash additive under mode II and III loading.

Lack of comprehensive data describing the effect of siliceous fly ash additive on the fracture processes in structural concretes caused that **the main reason** to raise the subject of scientific work was experimental determination of fracture toughness of concrete—in which a portion of the binder was replaced with siliceous fly ash—under all three modes fracture.

The second reason is that encouraging the commencement of work on the analysis of the fracture processes in concretes with the siliceous fly ash additive was closely associated with the phenomenon of the variable dynamics of increasing the strength of such materials in the process of curing [24]. Due to the slow course of the pozzolanic reaction that has a direct influence on the mechanical properties of composites of this type, the increase in the strength at the initial stage of hardening is slow [25]. However, in the longer time of curing, the durability of cement with the siliceous fly ash reaches values exceeding the compressive strength of Portland Cement of the same strength class. In relation to the second reason of working on this particular subject of the scientific paper, efforts were made to analyze the concrete at early age and in a period exceeding 28 days:

- changes in compressive strength of concrete and
- changes in parameters of fracture mechanics of concretes.

All experiments were planned for two compositions of concrete mixture, with varying percentage of siliceous fly ash additive, which is often used in the cement industry. An assumption in the analysis in the target range resulted from the following fact. In tests, of which the results were presented in paper [8], fracture toughness under II mode fracture was analyzed in concretes with the siliceous fly ash additive in the amount of 10, 20, and 30% of weight of cement. On their basis, it was found that 10% siliceous fly ash additive has a minor impact on the value of parameter K_{IIc} causing it to increase by only 1%, while 20 and 30% additive of this waste significantly changes the fracture toughness. Therefore, all tests were conducted for concretes modified with the siliceous fly ash additive in the amount of 20 and 30% of weight of cement. The obtained results from experiments were compared to the values obtained for the reference concrete, which was a composite made with the use of cement CEM I.

2. Scope of the studies and methodology of the tests

2.1. Scope of the studies

In order to accurately understand the fracture processes in the concretes with the siliceous fly ash additive, an extensive program of laboratory tests on a wide range of diagnostic methods was developed. In the first stage, properties of siliceous fly ash used in concretes were characterized. In testing, an attention was paid to parameters of fly ash such as:

- chemical and phase composition,
- bulk density,
- specific surface area,
- pozzolanic activity, and
- natural radioactivity.

Then, for all planned testing of concrete, specimens with the siliceous fly ash additive were prepared. The fracture toughness tests to estimate critical stress intensity factors and the compressive strength tests were conducted with three types of concretes of different volume content of the FA additive (like in Refs. [8, 26–29]), that is:

- without the FA additive (FA-00),
- with 20% FA additive (FA-20), and
- with 30% FA additive (FA-30).

Concrete testing included:

- analysis of compressive strength parameter and
- analysis of parameters of fracture mechanics.

In the course of the work, macroscopic measurements of compressive strength and fracture toughness for all modes were conducted. A w/c ratio was constant in all tested concretes. In modified concretes, cement was replaced by fly ash by its weight. For each of the three composites, all experimental tests were conducted in six time periods, with the age of concrete being 3, 7, 28, 90, 180, and 365 days.

Advanced diagnostic equipment was used during the experiments. For the evaluation of fracture toughness, three separate test stands were organized (see Section 5). On the basis of the planned scope of works, associated with the realization of the academic achievement, complete repository of **detailed academic objectives** is as follows:

a. In the range of fly ash testing, the scientific objectives were:

- analysis of physicochemical properties of fly ash and
- evaluation of pozzolanic activity of fly ash.

b. In the range of fracture toughness, the scientific objectives were:

- experimental determination of the impact of siliceous fly ash additive on the critical stress intensity factor of concrete under I, II, and III mode fracture, between the 3rd and 365th days of curing and
- determination of particular concretes with generalized fracture toughness K_c.

Due to the transparency of this chapter, this section is limited only to provide general information related to the experiments. Detailed descriptions of the strength tests were presented in Section 4, while fracture toughness tests are presented in Section 5. The basic properties of used fly ash are described in section 3.

2.2. Materials used in the studies and methods used to investigate them

2.2.1. Materials used in the preparation of mixtures

In elaboration of a new concrete mixture having the fly ash additive, it was assumed that the total amount of binding material in the concrete should be constant. Therefore in this methodology, a certain portion of cement is replaced in the mixture by the same amount of the fly ash. The following materials were used for making the mixtures:

- Ordinary Portland Cement (OPC) CEM I 32.5 R,
- natural gravel aggregates of maximum grain size up to 8 mm,
- a pit sand with a maximum diameter of 2 mm,
- siliceous, class F, fly ash from local power plant is a result of energetic combustion of hard coal in the Puławy thermal-electric power station, and
- Basf Liquol BV-18 plasticizer (based on calcium lignosulphonates); 0.6% of binding material weight.

2.2.2. Binders investigations

The chemical compositions of the Ordinary Portland Cement as well as the fly ash were evaluated by X-ray fluorescence (XRF), whereas microstructure of these materials was evaluated by using Scanning Electron Microscope (SEM). The microstructural testing was carried out using a QUANTA FEG 250 at magnification from 200 to 80,000 times equipped with an energy dispersive Spectroscopy (EDS EDAX). The tests were performed in both the low and high vacuum. A bulk density in both materials was determined by the pycnometric method, whereas the specific surface area was determined by the Blaine method. The average value of the two tests was determined based on the three results.

2.2.3. Properties of the materials

The main parameters of the materials used in the studies are given below.

2.2.3.1. Ordinary Portland Cement

The physical parameters of Ordinary Portland Cement are collected in **Table 1**, whereas the chemical constituents are shown in **Table 2**.

The mineralogical compositions of Ordinary Portland Cement were analyzed by the Bogue method. They are listed in **Table 3**.

2.2.3.2. Aggregates

The basic properties of aggregates are shown in **Table 4**, whereas the particle size distribution in **Table 5**.

An attempt was made to select the optimum proportion of different-sized aggregates in such a way as to be contained in the most advantageous area between limiting grain size distribution curves. The recommendations used were based on the German standard DIN 4226-1.

2.2.3.3. Admixture

In order to improve the workability of concrete mixtures, plastifying admixture composed on the basis of calcium lignosulfonate with a density of 1.16 g/cm^3 and the dosing range of

Physical parameters of cement		**Average values**
Specific surface area according to Blaine (cm^2/g)		3280
Bulk density (g/cm^3)		3.11
Initial setting time (min)		207
Final setting time (min)		298
Compressive strength (MPa)	After 2 days	23.3
	After 28 days	50.0
Volume change (mm)		0.56

Table 1. Physical parameters of Ordinary Portland Cement CEM I 32.5 R.

Constituent	Contents (% mas.)
SiO_2	21.37
Al_2O_3	5.02
Fe_2O_3	2.40
CaO	63.95
MgO	2.47
SO_3	3.0
Na_2O	0.18
K_2O	0.91
Cl	0.057
Insoluble residues	1.11
Loss of ignition	1.24

Table 2. Chemical composition of the Ordinary Portland Cement CEM I 32.5 R.

C_3S	C_2S	C_3A	C_4AF	$CaSO_4$ (gypsum)
60.69	15.82	9.24	7.28	5.10

Table 3. Mineral components of the Ordinary Portland Cement CEM I 32.5 R (%).

Property	Unit	Aggregate type	
		Fine aggregate—sand	Coarse aggregate—gravel
Specific density	g/cm^3	2.60	2.65
Bulk density	g/cm^3	2.20	2.25
Compressive strength	MPa	33	34
Modulus of elasticity	10^2 MPa	330	330
Sand point for mix	%	40.7	

Table 4. The basic properties of aggregates used in the study.

0.1–1.0% of mass of cement was used. The plasticizer is used in an amount of 0.6% of mass of the binder.

2.3. Mixing casting and specimens curing

A mix proportion of the concrete compositions are presented in **Table 6**. All mixtures had the same water-binding material indicator $w/b = 0.4$. The cast specimens were covered with a polyurethane sheet and damped cloth. They were placed in 20 ± 2°C chamber. After 2 days, all

Fraction (mm)	Content of aggregates fraction (%)		
	Sand	Coarse aggregate	Mix
0–0.125	2.9	0.7	1.7
0.125–0.25	14.8	0.4	5.6
0.25–0.5	41.1	0.4	15.3
0.5–1.0	32.7	1.6	12.4
1.0–2.0	4.5	6.9	5.7
2.0–4.0	4.0	19.9	13.9
4.0–8.0	0.0	63.1	40.2
8.0–16.0	0.0	7.0	5.2
Sand point	96.0	10.0	40.7

Table 5. The particle size distribution of the aggregates used.

Concrete	Cement (kg/m^3)	Fly ash (kg/m^3)	Water (kg/m^3)	Sand (kg/m^3)	Coarse aggregate (kg/m^3)	Plasticizer (kg/m^3)
FA-00	352	0	141	676	1205	2
FA-20	282	70	141	676	1205	2
FA-30	246	106	141	676	1205	2

Table 6. Mix proportion of the concrete compositions.

specimens were demolded. Then, they were kept for the first 14 days in a chamber with a moisture-saturated atmosphere. During the next days to the study after 28, 90, 180, and 365 days, specimens were cured in laboratory conditions (20 ± 2°C). In case of specimens tested after 3 and 7 days, they were removed from the water at least a few hours before the study. After a suitable period of curing, the compressive strength tests and other basic tests were carried out.

2.4. Specimens used in the studies

When deciding about the number of specimens for experiments, attempts were made to reach a compromise between the cost of their preparation as well as the minimum amount necessary to guarantee reliable statistical values and the ability to generalize the results obtained from specimens on the entire population of concrete. Therefore, in all macroscopic tests, average values of the conducted experiments were assessed on the basis of results obtained for six specimens. Assortment of specimens for compressive strength and fracture toughness tests of concrete, for each of the mixtures in all time periods, was as follows:

- 6 cubic specimens (150 mm) for testing the compressive strength—f_{cm},
- 6 beams (80 × 150 × 700 mm) with one initial crack for testing fracture toughness under mode I fracture—K_{Ic}^{S},

- 6 cubic specimens (150 mm) with two initial cracks for testing fracture toughness under mode II fracture—K_{IIc}, and
- 6 cylindrical specimens with a diameter of 150 mm and a height of 300 mm having an initial circumferential notch, for testing fracture toughness under mode III fracture—K_{IIIc}.

2.5. The concept of analysis of results obtained in experimental tests

When compiling the results of strength and fracture toughness tests, tables for further analysis for each type of the conducted experiments included average values and standard deviations—δ. Furthermore, graphs presenting the summary of the given parameter for all series of concrete, in all time periods, were prepared for average values in particular tests. The relationships of the analyzed indicators as a function of the age of concrete were shown graphically in order to better illustrate the changes occurring in concretes during their curing. Percentage changes of parameters, during curing of composites, were also shown, taking the value obtained after 365 days as 100%.

Results of the fracture toughness, for all modes of cracking, and generalized fracture toughness are summarized in **Table 11**.

3. Characteristics of used siliceous fly ash

3.1. Microstructure of cementitious materials

Figure 2 shows the morphologies and the particle sizes of the cementitious materials (cement and fly ash) by SEM observations. In order to accurately compare the microstructure of both fillers in the SEM studies, the same scale and magnifications were used (**Figure 2**).

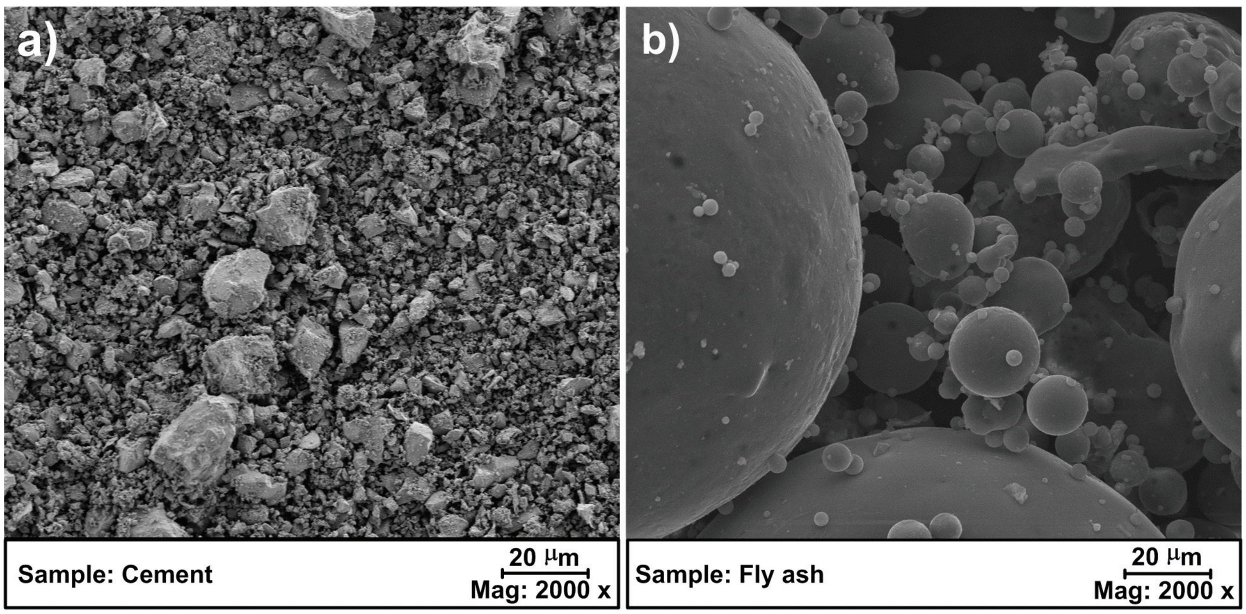

Figure 2. SEM images of cementitious materials: (a) cement and (b) fly ash.

Other interesting characteristic pictures of a typical grains and microstructures of the used fly ash at various magnifications were shown in paper [8]. On the basis of analysis of SEM images (**Figure 2(b)**), it can be concluded that fly ashes are characterized by oval regular shapes of fine particle size.

3.2. Chemical composition of the fly ash

The chemical constituents of the fly ash are shown in **Table 7**. The fly ash is a class F with 85.09% of SiO_2 + Al_2O_3 + Fe_2O_3, 0.65% of SO_3, and 3.2% of loss of ignition (LOI) meeting the requirement of ASTM C618.

Constituent (wt. %)	**SiO_2**	**Al_2O_3**	**CaO**	**MgO**	**SO_3**	**Fe_2O_3**	**LOI**
Fly ash	50.96	25.88	2.15	2.6	0.65	8.25	3.2

Table 7. Chemical composition of the fly ash.

Parameter	**Average values**
Specific surface area according to Blaine (cm^2/g)	3640
Bulk density (g/cm^3)	2.14

Table 8. Physical parameters of the fly ash.

3.3. Physical parameters of the fly ash

The main physical parameters of the fly ash are shown in **Table 8**.

Physical properties of the cement and the fly ash, and **Figure 2** suggest that the fly ash is finest, followed by the cement. Small particle size of the fly ash has a beneficial effect on compressive strength, durability, and permeability of concretes with these additives [30].

3.4. Mineral composition of the fly ash

The crystalline phases of the fly ash were identified by X-ray diffraction (XRD) analysis [8]. The crystalline phases of the fly ash were identified by XRD patterns. Randomly oriented powder specimen (about 1 g weight) for XRD analysis was prepared. XRD graphs were obtained at room temperature by a PANalytical X'Pert PRO MPD diffractometer (with the PW 3050 goniometer), Cu lamp, and graphite monochromator. The diffractogram from 5° to 65° in the $2\theta$and scanning rate was at 0.05° intervals. PANalytical X'Pert HighScore and the ClayLab ver. 1.0 software were used to process diffraction data. The identification of mineral phases was based on the PCPPDFWIN ver. 2.1 database formalized by the JCPDS-ICDD.

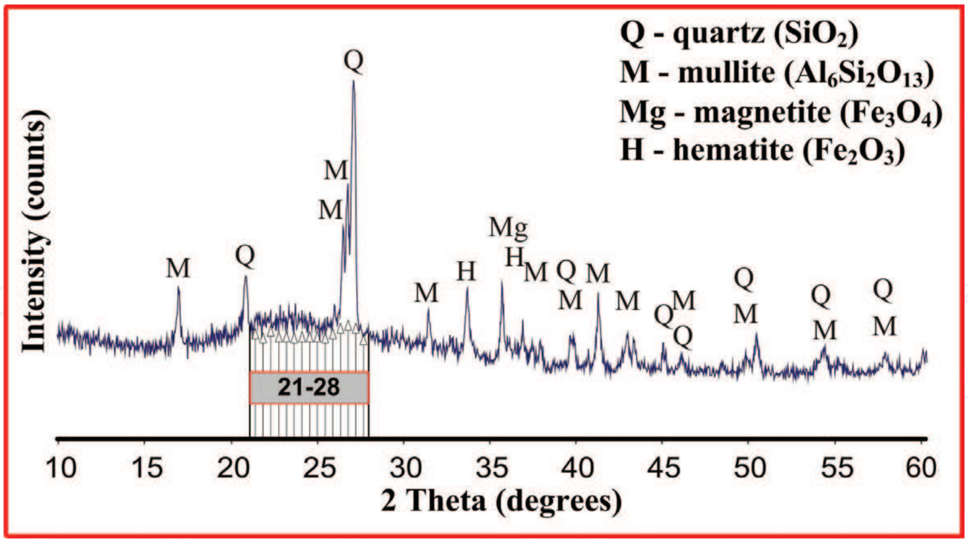

Figure 3. XRD pattern of the fly ash.

The X-ray diffractogram of the fly ash (**Figure 3**) shows that there are, beside glass, four major crystalline components in the phase composition of the fly ash: quartz (SiO_2), mullite ($Al_6Si_2O_{13}$), magnetite (Fe_3O_4), and hematite (Fe_2O_3). On the diffractogram, a characteristic increase in intensity at the angle ranging from 21 to 28° 2θ can be observed as well as relatively low intensity of the peaks of quartz and mullite phases. This image of the phase composition of the fly ash indicates their fine particle size and a high-strength activity index.

3.5. Pozzolanic activity of the fly ash

The applicability of the fly ash depends mainly upon its pozzolanic activity. In order to assess the pozzolanic activity of the used FA were selected physical method, according to EN 4501-1 standard. EN 450-1 defines as a standard pozzolanic activity index of the fly ash the strength activity index which is the ratio (in %) of the compressive strength of mortar containing 75 wt% of cement CEM I 42.5R, 25 wt% of the fly ash and cement mortar without addition. According to EN 450-1 standard, the pozzolanic activity index of the fly ash is determined after 28 and 90 days of hydration. The compressive strength of cement is measured according to procedure described in EN 196-1 standard, using 40 × 40 × 160 mm prisms of the mortar. The water-to-solid ratio in mortars is 0.5. According to the EN 450-1 standard, the acceptable level of pozzolanic activity is attained when the 28-day compressive strength of the fly ash and cement mortar constitutes 75% of the value for the control sample and after 90 days—85%, respectively.

Because compressive strength tests and fracture toughness test were conducted in six time periods in the range between 3 and 365 days, pozzolanic activity index was also determined at the same time intervals, that is, after 3, 7, 28, 90, 180, and 365 days. **Table 9** shows the average values of the strength activity index, whereas **Figure 4** shows how it looked its growth rate.

Age (days)	Pozzolanic activity index (%)
3	56.73
7	68.36
28	92.13
90	111.84
180	116.72
365	123.53

Table 9. The assessment of pozzolanic activity by EN-450-1 standard.

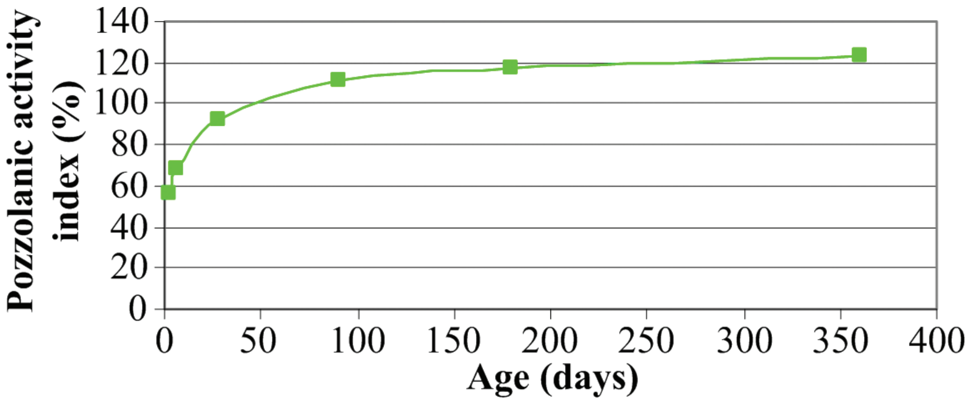

Figure 4. Pozzolanic activity index vs. age of mortar.

A line connecting points on the graph (**Figure 4**) shows a gradual, non-linear increase of the activity of the fly ash between 3rd and 365th days of curing of samples. After 3 days, the strength activity index is very low, which is certainly due to the slow growth of the reaction products after such a short period of curing. According to Lee et al. [31], siliceous fly ash after 1 day is still inactive, and according to Ogawa et al. [32], pozzolanic reaction in this material begins in 1–3 days. Also Fraay et al. [33] note that up to 1 week, the fly ash does not show a distinct amount of pozzolanic reactivity.

Based on the strength activity index (**Table 9**), it can be concluded that the initial effect of the pozzolanic reaction (consisting in strengthening the matrix) in the composites, which include the addition of siliceous fly ash, appears until after 7 days of curing. This is also confirmed by other studies [34]. After this time, it can be seen a significant increase in the activity of the material—more than 20%. In the period between the 7th and the 28th days is already visible intense increase in the strength of mortars with fly ash and the activity index compared to the value of weekly increases by almost 35%. A significant increase in the analyzed parameter is visible even between the 28th and the 90th days of curing of samples. In a further period of time, between 3 and 6 months and after a year, there has been a slight increase of the pozzolanic activity of fly ashes (by a few percent). It can be concluded that after 6 months, the ongoing reactions stabilizes. Nevertheless, the reactions still exist even after 365 days [34].

As a result of the study, it can be concluded that the pozzolanic activity of the fly ash meets the requirements of EN 450-1 standard. Strength activity index was 92.13% at 28 days and 111.84% after 90 days, which means that it exceeds significantly the minimum values specified in the requirements of the EN-450-1 standard.

3.6. Natural radioactivity of the fly ash

Natural radioactivity of the fly ash and concrete made with their addition was analyzed based on concentration of radioactive elements (potassium, radium, and thorium), radioactivity coefficients f_1 and f_2, and the radiation dose [35]. The study of natural radioactivity of used fly ash shows that these materials are not hazardous from the radiological point of view. Both concentration of radioactive elements and radioactivity coefficient and radiation dose were contained within acceptable limits [35].

4. Compressive strength analysis

The uniaxial compression strengths were tested using a compression machine (Walter + Baiag) with a maximum load of 3000 kN. The loading rate of compressive strength test was controlled between 0.5 and 0.8 MPa/s. The compressive strengths f_{cm}, were tested with application of cubic specimens (described in Section 2.4) according to the standards of series EN 12390.

Table 10 summarizes the results from compressive strength tests of concretes, for particular periods of curing, while **Figure 5** presents relationships f_{cm}, as a function of the age of concrete.

Fly ash additive caused a clear decrease of compressive strength in concretes analyzed at an early age, that is, after 3 and 7 days. According to **Table 10**, value f_{cm} after 72 h of curing was almost 8 and exactly 10 MPa higher in concrete without the fly ash additive in comparison to FA-20 and FA-30, respectively. After a week, the differences between the reference and modified concretes were only 3 MPa. According to Ref. [26], in which a more detailed analysis

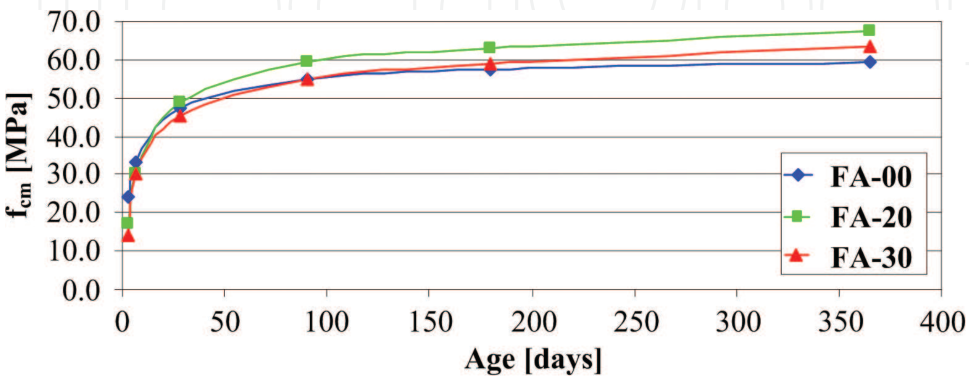

Figure 5. Compressive strength of analyzed concretes as a function of curing time.

Concrete	Age (days)	$f_{cm} \pm \delta$ (MPa)
FA-00	3	24.23 ± 2.60
	7	33.18 ± 2.57
	28	47.51 ± 2.55
	90	55.13 ± 2.51
	180	57.22 ± 2.48
	365	59.25 ± 2.46
FA-20	3	16.95 ± 3.05
	7	30.12 ± 3.03
	28	48.96 ± 3.02
	90	59.35 ± 2.80
	180	62.81 ± 2.52
	365	67.29 ± 2.35
FA-30	3	14.23 ± 3.59
	7	30.06 ± 3.57
	28	45.10± 3.55
	90	55.11 ± 3.10
	180	58.83 ± 2.86
	365	63.27 ± 2.50

Table 10. Compressive strength of concretes.

of changes of strength parameters in concretes with the fly ash additive at an early age is presented, clear disproportions in the obtained results occur within 14 days of curing. After 3 weeks, 20% fly ash additive strengthens the structure of concrete composites to the extent that the values of f_{cm} are slightly higher in this type of concrete in comparison to FA-00. Also, after 28 days and in subsequent time periods, FA-20 had by far the highest strength, which was probably due to the rapid increase of pozzolanic reaction products after a longer time of curing. Concrete with a larger amount of fly ashes was characterized by the lowest strength in the period up to 3 months. After a half year of curing, value of f_{cm} for this composite was higher in comparison to concrete without the additives; however, it still lower in comparison to FA-20. A further increase of strength of FA-30 caused that after a year strength of this material was 4 MPa higher f_{cm} in comparison to FA-00 and lower by the same value in comparison to FA-20.

The conducted own tests have shown that compressive strength of concrete was increasing with time. The growth dynamics of this parameter, however, differed significantly in particular types of analyzed composites. This can be easily observed by comparing relative changes of compressive strength over time, which is shown in **Figure 6**.

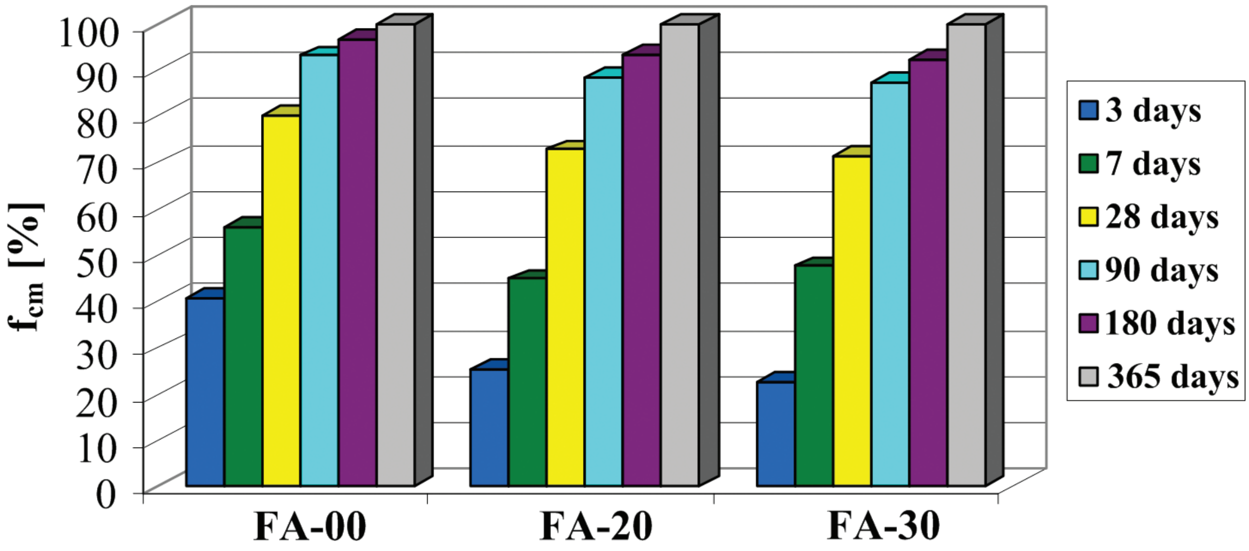

Figure 6. Relative changes of compressive strength over time.

After the first measurement (3 days), concrete FA-00 had more than 40% of the annual compressive strength, whereas in concretes with fly ash additives, this strength did not even had 30% of the final strength. After a week, concrete, without the fly ash additive, was characterized by a higher relative strength, however, in the analysis of obtained values, a clearer dynamics of strength increase in composites with fly ashes was observed. Although reference concrete had already more than 50% of its 365-day strength, which was a result 10% better in relation to concretes with additives, a greater increase of f_{cm} was observed in concretes with fly ashes between 3rd and 7th days of curing. Also, between 7th and 28th days of curing, concretes with additives are characterized by significant increments of f_{cm} (amounting to: FA-20—50%, FA-30—62%), which correlates with the activity of pozzolanic fly ashes obtained with the use of physical method in accordance with EN 450-1 (**Table 9**, **Figure 4**). Clear growth trends of compressive strength, in modified composites, were observed even after 90 days after the start of tests. Both 20 and 30% fly ash additives caused increase of f_{cm} within 2 months by more than 20%, which was a result 6% better in relation to FA-00. After half a year in all types of composites, the increase of compressive strength was small and was 6% for modified concretes and only 3% in the reference concrete. A similar trend was also observed in relation to 1-year concretes where the strength increase in concretes with fly ashes was 7% and was higher by half in comparison to the results obtained for FA-00.

5. Fracture toughness tests

5.1. Fracture toughness tests during mode I loading

The testing of Mode I fracture toughness was performed according with the draft guidelines of International union of laboratories and experts in construction materials, systems and structures (RILEM) recommendations [36]. The fracture toughness of the composites with the fly ash addition was determined based on the experimental results of critical stress intensity factor—K_{Ic}^{S}.

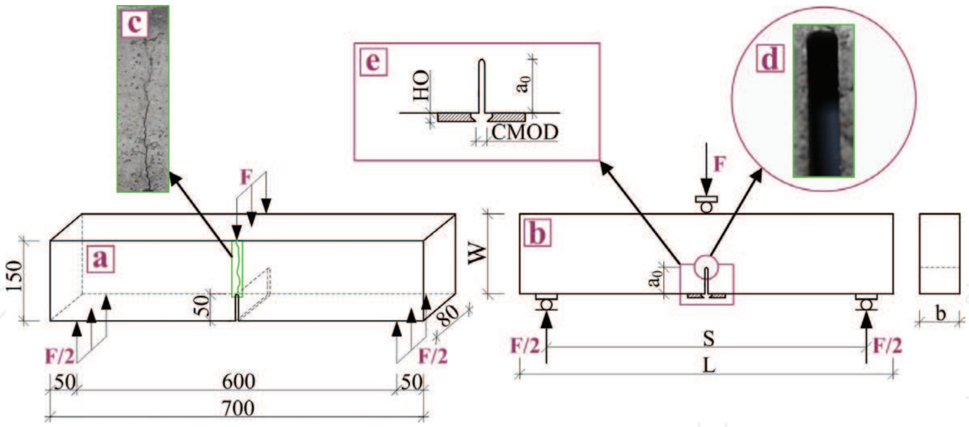

Figure 7. Specimen with (a) static scheme, (b) geometry, (c) exemplary crack paths, (d) blunted notch, and (e) details of calliper gauge holder.

To assess the fracture toughness of concrete, beams with the dimensions 700 × 150 × 80 mm (**Figure 7**) which had one initial centrally crack were used (see Section 2.4). The beams were subjected to three-point bending test. They were made in demountable bolted wooden forms. The assumed size of the initial crack in the beams was achieved by actually concreting flat steel plates, having a thickness of 3 mm. A special experimental stand was prepared as shown in [28]. All necessary results, needed to determine the critical stress intensity factors for concretes, were obtained with application MTS 810 testing machine. The width of the initial crack opening during the tests was measured using a crack opening sensor, that is, the MTS clip gage axial extensometer, which was placed on the clamping test grips (**Figure 7**).

The specimens placed in the experimental stand were subjected to cyclic loading process performed quasi statically. The maximum force (95% of the fracture load) in the one cycle was achieved in 5 min. Then, we start unloading process up to 0 kN. After that the cycles were repeated until the beams were broken into two parts. The whole cyclic deformation processes were described by the following curves:

- Force (*F*)—crack mouth opening displacement (*CMOD*) and
- Force (*F*)—deflection (*f*).

The obtained results (which are summarized in **Table 11**) allowed for determination of the fracture toughness K_{Ic}^{S} using the formula given in RILEM recommendations [35].

5.2. Fracture toughness tests during mode II loading

In order to assess the influence of the fly ash volume content on the shear fracture toughness in the considered concrete composites, compact shear specimens (CSS) were prepared for the basic tests [8, 16, 26, 27]. Therefore, cubes with 150 mm long edge and two fictitious cracks were used for the experiments (see Section 2.4). The target crack sizes were obtained by embedding in concrete the cubes being formed by two 4 mm steel sharpened flat plates. The experiments were performed for the loading scheme presented in **Figure 8**. Specimen

Concrete	Age (days)	$K^S_{Ic} \pm \delta$ (MN/m$^{3/2}$)	$K_{IIc} \pm \delta$ (MN/m$^{3/2}$)	$K_{IIIc} \pm \delta$ (MN/m$^{3/2}$)	K_c(MN/m$^{3/2}$)
FA-00	3	0.58 ± 0.08	2.26 ± 0.38	1.36 ± 0.19	2.70
	7	0.79 ± 0.09	3.18 ± 0.41	1.88 ± 0.18	3.78
	28	1.06 ± 0.10	4.24 ± 0.40	2.49 ± 0.20	5.03
	90	1.21 ± 0.08	4.93 ± 0.35	2.92 ± 0.19	5.86
	180	1.26 ± 0.06	5.12 ± 0.30	3.06 ± 0.17	6.10
	365	1.30 ± 0.05	5.31 ± 0.24	3.19 ± 0.14	6.33
FA-20	3	0.40 ± 0.09	1.48 ± 0.34	0.92 ± 0.19	1.79
	7	0.73 ± 0.09	2.93 ± 0.40	1.75 ± 0.21	3.49
	28	1.09 ± 0.11	4.39 ± 0.51	2.60 ± 0.24	5.22
	90	1.31 ± 0.10	5.33 ± 0.41	3.16 ± 0.22	6.33
	180	1.39 ± 0.07	5.70 ± 0.32	3.36 ± 0.19	6.76
	365	1.48 ± 0.06	6.14 ± 0.29	3.59 ± 0.18	7.26
FA-30	3	0.30 ± 0.08	1.05 ± 0.25	0.68 ± 0.16	1.29
	7	0.62 ± 0.08	2.40 ± 0.31	1.41 ± 0.19	2.85
	28	0.93 ± 0.10	3,65 ± 0.42	2.14 ± 0.21	4.33
	90	1.17 ± 0.09	4.68 ± 0.38	2.77 ± 0.20	5.56
	180	1.28 ± 0.08	5.16 ± 0.34	3.08 ± 0.18	6.14
	365	1.37 ± 0.07	5.58 ± 0.27	3.31 ± 0.17	6.63

Table 11. Fracture mechanics parameters of concretes.

dimensions in detail and exemplary crack paths, which were observed during experiment, were also shown in **Figure 8**.

The tests were performed on the MTS 810 servo-hydraulic testing machine, like in the studies during mode I loading. Fracture toughness of the concretes was determined on the basis of analysis of critical stress intensity factor K_{IIc}. This factor was determined according to the formula proposed by Watkins [37], whereas average test results are summarized in **Table 11**.

5.3. Fracture toughness tests during mode III loading

In order to determine the fracture toughness K_{IIIc}, cylindrical specimens with a diameter of 150 mm and a height of 300 mm with an initial circumferential notch of 2 mm thickness (made in the half-height) were tested. The depth of the notch was equal to ¼ of the cylinder diameter. Initial notches in the specimens were created during their formation by application of the two semi-circular steel inserts placed in the half-height of specially prepared cylindrical forms [29].

The experimental stand (**Figure 10**) for the fracture toughness test at the mode III fracture consisted of the cylindrical specimen with the initial notches, steel plates, and screws with washers securing the specimen in the press holders, tension-torsion MTS testing machine [29].

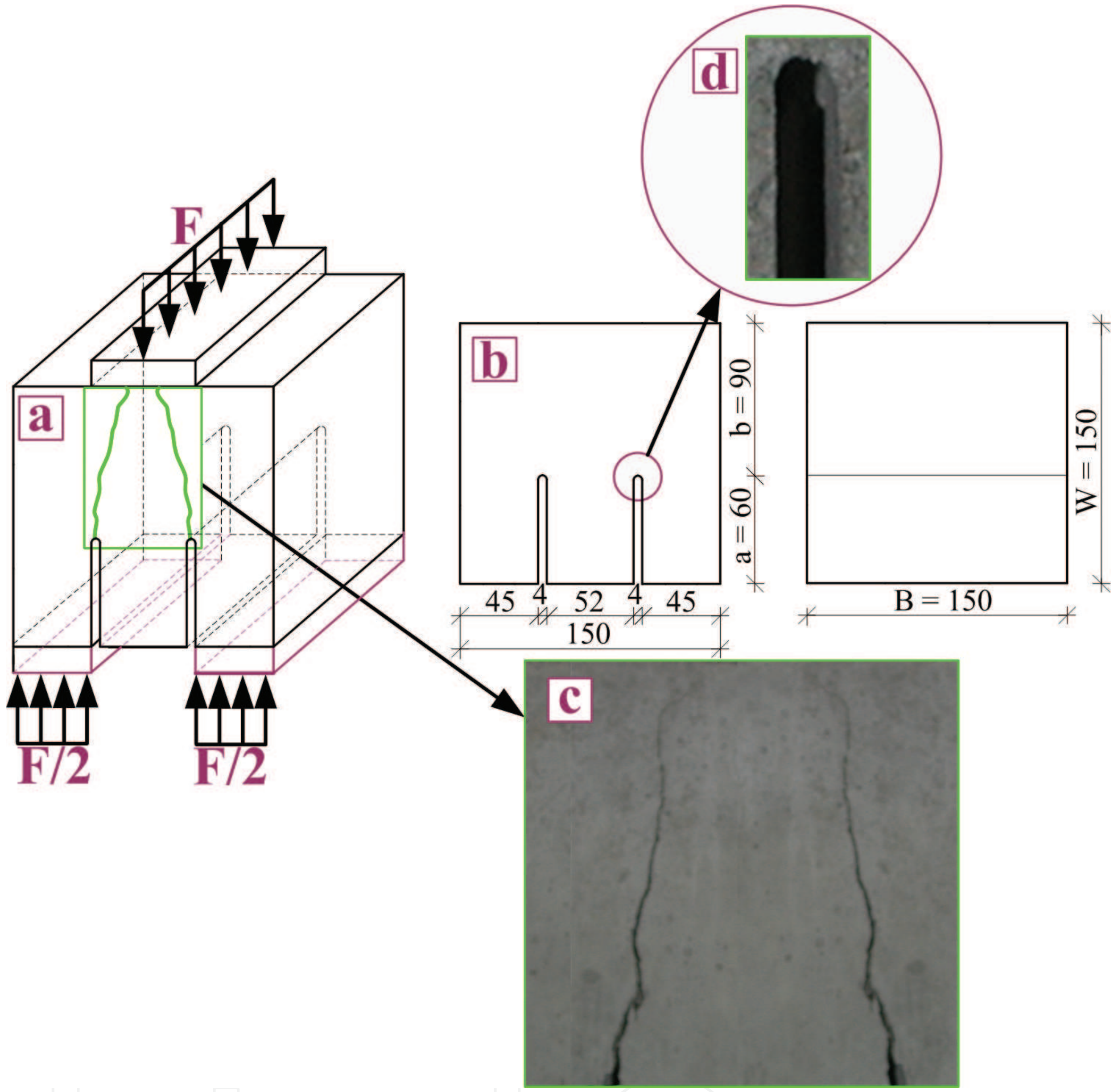

Figure 8. Specimen with (a) loading conditions, (b) dimensions (in mm), (c) exemplary crack path, and (d) blunted notch.

To mount the specimens in the grips of the torsion testing machine, two types of circular steel plates were designed and manufactured. During forming, the bottom plates of 15 mm thickness (**Figure 9**) were anchored in the specimens on their top and bottom with 6 bolts M12/65. Then, top plates of 10 mm thickness (**Figure 9**) were bolted to these plates with 6 bolts M12/20. Top plates hold the specimen directly in the grips of the torsion testing machine from the top and bottom with M28/70 bolts. The full device for testing the K_{IIIc} is shown in **Figure 9**.

Specimens were tested on the axial tension-torsion testing machine MTS 809 in accordance with the load diagram shown in **Figure 10**. The shear process of specimens was controlled by rotation angle ω assuming a small increase equal to 0.5°/min. Critical values of a torque and the rotation angle which corresponds to the failure of the specimens were reached after a few

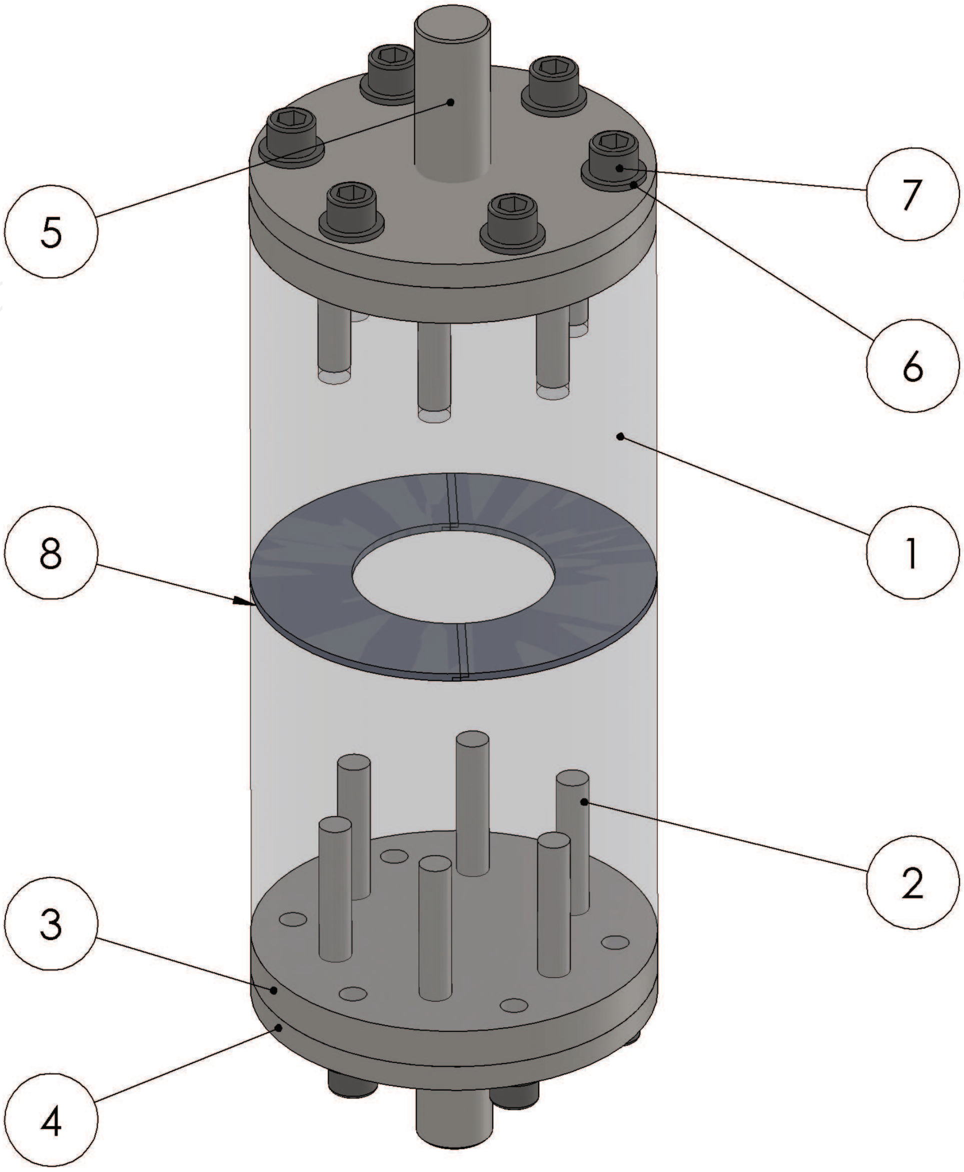

Figure 9. The device for fracture toughness test in the mode III fracture: 1—concrete specimen, 2—bolt M12/65, 3—bottom plate, 4—top plate, 5—bolt M28/70, 6—washers, 7—bolt M12/20, 8—insert.

minutes. On the basis of the experimental results, fracture toughness K_{IIIc} (**Table 11**) can be calculated, which was determined according to [38].

5.4. Determination of generalized fracture toughness

With the known values of K_{Ic}, K_{IIc}, and K_{IIIc}, it was possible to designate a generalized fracture toughness of the material K_c from the equation (1). On the basis of average values of critical stress intensity factors: K_{Ic}^S, K_{IIc} and K_{IIIc} in **Table 11** and **Figure 11** show results of generalized fracture toughness, for each of the analyzed concretes, in all time periods. In addition, **Table 11** summarizes the parameters of fracture mechanics obtained in tests for all three mode fractures.

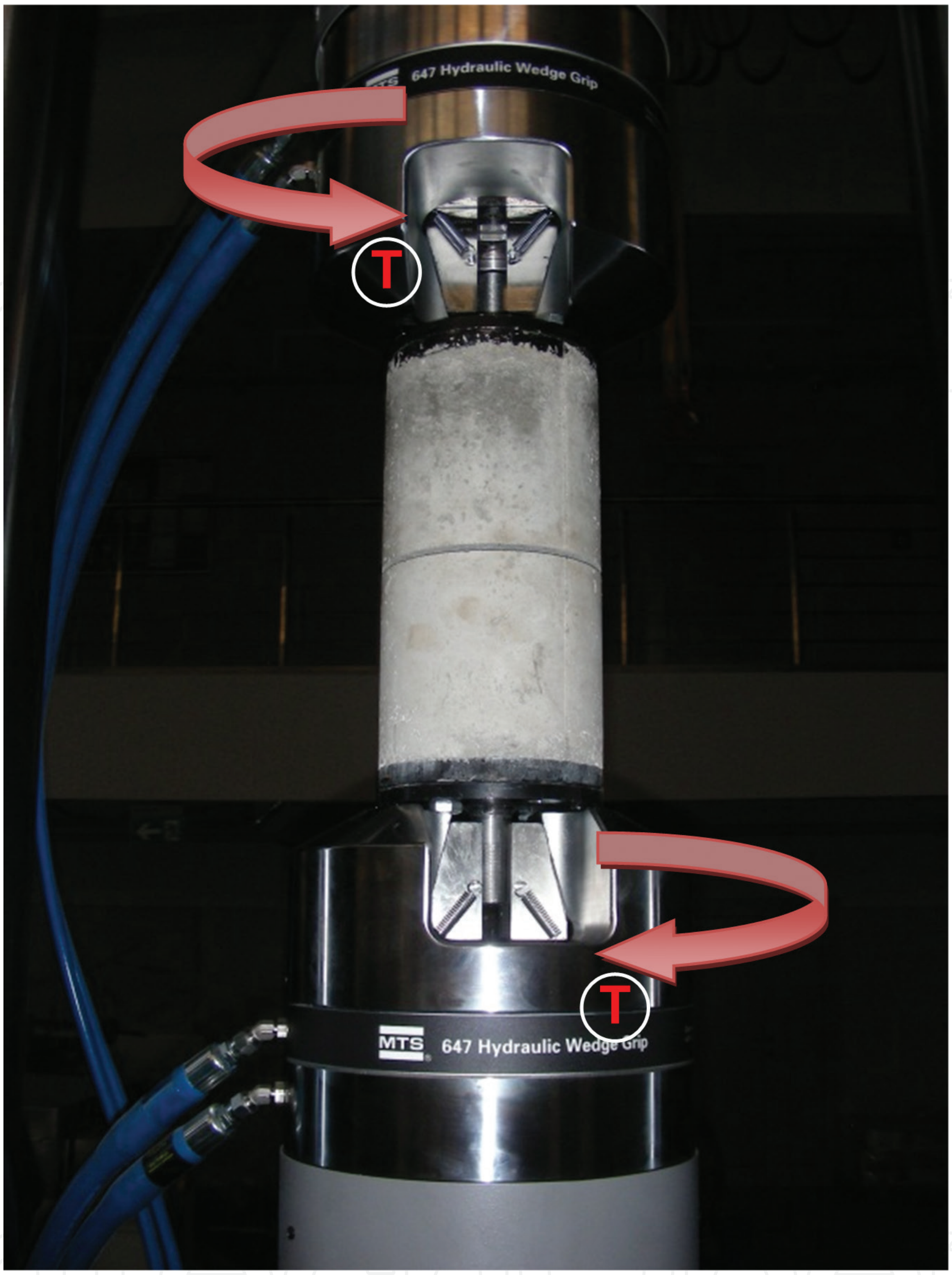

Figure 10. The experimental stand with the fixed specimen subjected to torsion; T, torque.

Based on the obtained results (**Table 11** and **Figure 11**), it can be concluded that after 3 and 7 days, the highest fracture toughness had concrete FA-00, while in the other four periods of time—FA-20. Concrete FA-30 had the lowest values of K_c in the range from 3 to 90 days, and after 180 and 365 days, the fracture toughness for this concrete is second to the results obtained for the composite with 20% siliceous fly ash.

The dynamics of changes of generalized fracture toughness in time (**Figure 12**) shows a very rapid increase of K_c after 3 and 7 days in the reference concrete and the delay of the processes

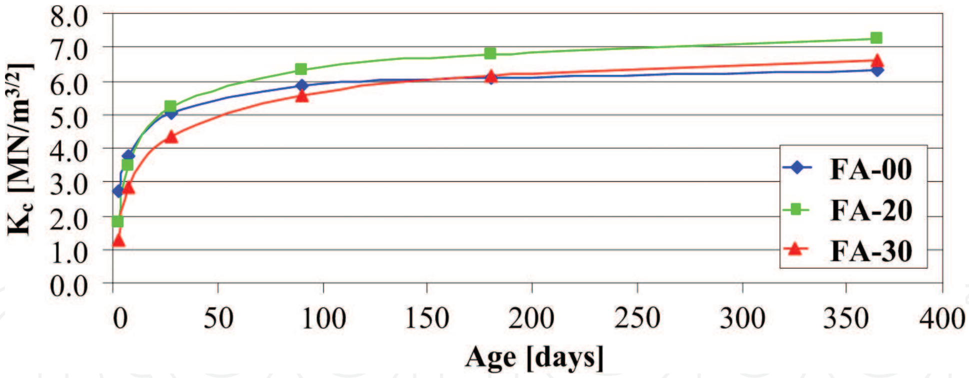

Figure 11. Generalized fracture toughness of analyzed concretes as a function of curing time.

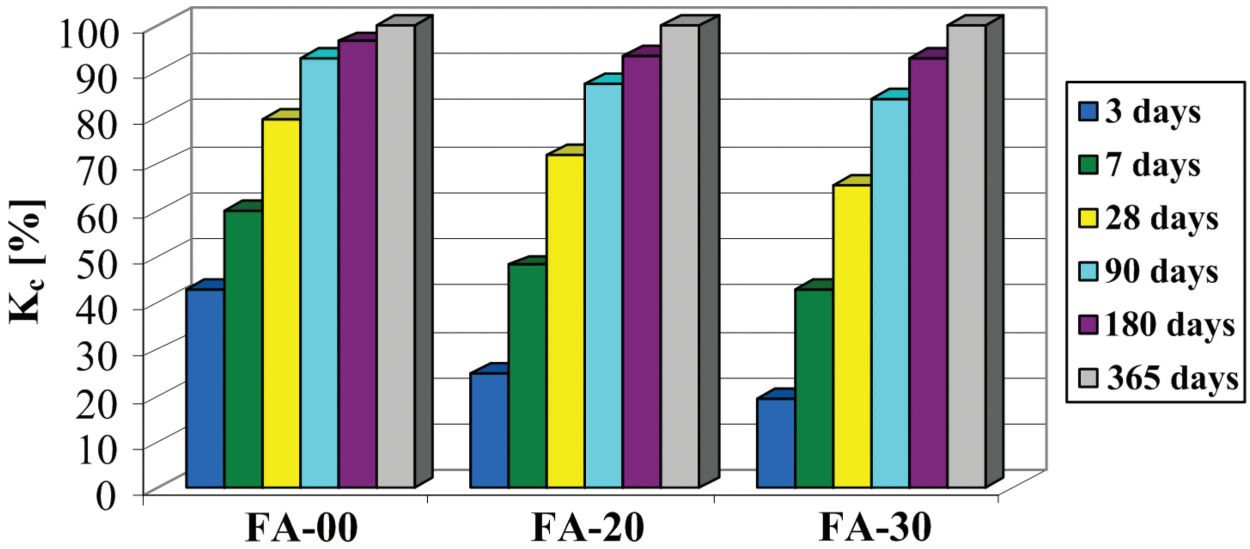

Figure 12. Relative changes of generalized fracture toughness over time.

for concretes with additives. Concrete FA-00 after 3 days reached more than 40% of the final value, while in the concretes with siliceous fly ash, it was less than 25% for FA-20 and less than 20% for FA-30. Also, after a week, large disproportions were noticed in the obtained results. The value of K_c in concrete FA-00 accounted for almost 60% of the annual result. At the same time, in concretes with siliceous fly ash, generalized fracture toughness was less than 50% K_c obtained for these concretes after 365 days. In the analysis of changes in values K_c in subsequent periods in which the tests were conducted, a steady increase of this parameter in comparative concrete as well as rapid increase in modified concretes is observed. As a result of the occurring processes, generalized fracture toughness exceeded 90% of the annual values after 6 months of curing in all of the analyzed concretes.

6. Discussion

Based on the results shown in **Table 11**, it can be concluded that siliceous fly ash additive clearly changes its generalized fracture toughness. At early age, the structure of materials with these components significantly weakens. With the 20% fly ash additive, parameter K_c after 3 days was 33.7% lower while after 7 days, it was 7.67%, in relation to values obtained for FA-00. More evident change of generalized fracture toughness resulted in an increase of the amount of the cement substitute in concrete by 10%. This resulted in a further decrease of K_c by almost 20% in 3- and 7-day concretes in comparison to results for comparative concrete. Another analysis of K_c already done for mature concrete also reflects the negative impact of 30% fly ash additive, but in relation to FA-20/28, it shows a completely different trend. Twenty percent siliceous fly ash additive after a month of curing causes an increase in fracture toughness by 3.78%, while 30% additive causes a clear drop of K_c by 13.92%. This shows that after 28 days, the 20% fly ash additive causes beneficial effects resulting from the pozzolanic reaction, whereas above this value, there is still a decrease of mechanical parameters in modified concretes.

The analysis of generalized fracture toughness in subsequent periods of time indicates that for 90-day concretes, another increase of K_c in concretes with fly ashes can be observed to the extent that the value of this parameter obtained for FA-20 is greater by 8% in relation to the value obtained for FA-00. When comparing the value of K_c for the reference concrete and FA-30, it is concluded that K_c is still greater in FA-00. However, the difference is slight and is only 5%. For concretes tested after 180 and 365 days, beneficial effect of fly ash additive is in both smaller and larger percentages of its content. Generalized fracture toughness in concretes with ashes already exceeds the values obtained for FA-00/180 and FA-00/365 by a few and more than 10% for FA-20/180 and FA-20/365 as well as 1 and 5% with respect to FA-30/180 and FA-30/365.

When comparing the effect of siliceous fly ashes on values of generalized fracture toughness (**Table 11**) and compressive strength of concretes (**Table 10**), distinct similarities in the effect of used mineral additives on the results obtained in both tests can be observed.

7. Conclusions

On the basis of comprehensive fracture toughness tests, in which a portion of the binder was replaced with active pozzolanic siliceous fly ashes, it can be concluded that:

- the siliceous fly ash additive in the amount of 20 and 30% of mass of cement significantly affects the change of fracture toughness in tension, shear, and torsion,
- obtained parameter values of fracture mechanics depend on the age of concrete,
- siliceous fly ash additive in the amount of up to 30% of mass of cement drastically reduced the fracture toughness at early age,
- 20% siliceous fly ash additive ensures high fracture toughness in mature concretes,

- concretes with 30% siliceous fly ash additive are characterized by highest dynamic increase of the parameter K_c,
- after 180 and 365 days, fracture toughness of FA-30 concrete is higher in comparison to the values obtained for FA-00 concrete.

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