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Fracture parameters of fine-grained composites based on the alkali-activated slag

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Abstract. The paper deals with the experimental determination of fracture parameters of the composites based on the alkali-activated slag (AAS) at different ages. The main aim of the experimental investigation was to verify the effect of the addition of shrinkage reducing admixture (SRA) on the overall progress of length changes and fracture properties during AAS composites ageing. Two AAS composites, which differed only in the presence of SRA, were prepared for the purpose of experiments. The mechanical fracture parameters were determined using evaluation of fracture tests carried out on 40 × 40 × 160 mm beam specimens with an initial central edge notch. The monitored parameters were modulus of elasticity, effective fracture toughness and specific fracture energy. These parameters were determined at the age of 3, 28 and 90 days. The obtained results are supplemented by the results of relative length changes measurements performed on both studied composites.

1. Introduction

Although cement based composites are the most commonly used quasi-brittle structural materials in the building industry in the world, the environmental aspects related with their production and using of cement and concrete are increasingly being discussed. Their manufacturing process significantly contributes to the global emissions of CO₂ [1]. Therefore, there has been an increasing effort to develop and use some alternative binders instead of ordinary Portland cement (OPC) during the recent decades. The alkali-activated aluminosilicate materials are one representative of such type of binders. These binders are created through the mixing of some non- or poorly-crystalline aluminosilicate based material, such as blast furnace slag (BFS) or fly ash (FA), with an alkaline activator (hydroxides, carbonates or the most preferably silicates) and water [2, 3]. Type and dosage of the activator as well as the way of curing have a significant effect on the hydration process and resulting mechanical properties [4]. The major disadvantage of composites based on alkali-activated matrix (AAM) is an increased shrinkage during hardening period, caused by both autogenous and drying shrinkage, which finally results in volume contraction, microcracking and deterioration of tensile and bending properties [5]. For the purpose of reduction of the negative effect of the high shrinkage on the development of physical and mechanical characteristics of alkali-activated composites, shrinkage reducing admixtures (SRA) or fibres are the mostly used [6, 7].
Because there is not a sufficient amount of available information about fracture behaviour of composites based on AAM, the attention of this paper is besides others also paid on mechanical fracture parameters determined based on three-point bending fracture test evaluation.

2. Material and specimens

In the experiment described in this paper, two fine-grained composites based on alkali-activated slag (AAS) were tested. The difference between mixtures was only in the presence of SRA. The experimental and numerical analysis was aimed at the investigation of the effect of SRA on the overall progress of length changes and fracture properties during AAS composites ageing. Ground granulated blast furnace slag with Blaine fineness around 400 m$^2$/kg was activated by water-glass with silicate modulus of 2.0 to produce alkali-activated binder. The amount of water-glass was adjusted to provide 10% of Na$_2$O by slag weight and the water-to-slag ratio was 0.42 including extra added water and water present in water-glass. To prepare fine-grained composites, the silica sand with maximum grain size of 2 mm was mixed with slag and activating solution in the dose three times higher than the slag weight. Commercially produced SRA, originally developed for Portland cement based concretes (Stachement AC 600), was chosen, its amount in second mixture was 2% by weight of slag. The sets of specimens were labelled as follows: AAS and AAS_SRA, specimens without and with SRA, respectively.

The AAS composites were mixed by a laboratory mixer with a mixing speed of 30 revolutions per minute and then were cast into polypropylene moulds to prepare prismatic specimens with dimensions of 40 × 40 × 160 mm intended for the fracture test. After 72 hours, specimens were demoulded and stored until the start of experiments. Curing conditions (ambient temperature of 21 ± 2°C and relative humidity of 60 ± 10%) were the same before and after demoulding. Such severe curing conditions were selected to intensify shrinkage and to evaluate the effect of the used SRA. In total, 18 specimens were made from each mixture; six specimens were tested at each investigated age of specimens. The fracture parameters were determined at the age of 3, 28 and 90 days.

Furthermore, length changes of both investigated mixtures on the specimens with dimensions of 100 × 60 × 1000 mm were continuously determined using apparatus with movable front walls. Free movement of specimens in the moulds was ensured by polyethylene foam mat (MIRELON). Curing conditions were the same as were described above for specimens intended for fracture tests. Length changes measurement continued also after demoulding of these specimens (72 hours) up to approximately 90 days. Then the length changes measurement was finished and additional specimens with nominal dimensions 40 × 40 × 160 mm were cut from these bigger specimens and also subjected to the fracture tests. The specimens obtained in this manner are designated as “cut”.

3. Fracture tests

All above mentioned test specimens were provided by an initial central edge notch with depth about one third of specimen height and subsequently loaded in three-point bending fracture test configuration. The tests were performed using the mechanical testing machine Heckert FP 10/1 which allows loading with controllable speed of displacement increment. For the purpose of this experiment, loading speed was set to value of 0.02 mm/min. The displacement increment loading of the specimen allows recording the load vs. displacement (deflection in the middle of span length) diagrams using an inductive sensor connected to HBM SPIDER 8 data logger during the testing. The measured $L$–$d$ diagrams are introduced in Figure 1. One diagram for each mixture and investigated age of specimens is shown for illustration.

The first (almost linear) parts of the recorded $L$–$d$ diagrams were used for the estimation of the elasticity modulus value. Furthermore, the effective fracture toughness values were determined using the Effective Crack Model [8], which combines the linear elastic fracture mechanics and crack length approaches. The work of fracture and afterwards specific fracture energy values were obtained from the $L$–$d$ diagrams according to the RILEM method [9].
Figure 1. The selected $L$–$d$ diagrams.

4. Results
The mean values and standard deviations (usually determined from 6 independent measurements) of selected mechanical fracture parameters obtained from the recorded $L$–$d$ diagrams using above described approach [8, 9], i.e. modulus of elasticity, effective fracture toughness and specific fracture energy, are summarized in Figure 2, 3 and 4. The obtained values are introduced for specimens at the age of 3, 28 and 90 days.

Figure 2. The values of modulus of elasticity of alkali-activated fine-grained composites.
Figure 3. The values of the effective fracture toughness of alkali-activated fine-grained composites.

Figure 4. The values of the specific fracture energy of alkali-activated fine-grained composites.

All investigated mechanical fracture parameters show unexpected decrease in their values at the age of 28 days. In the case of AAS specimens, the values decreased to less than 1/10 in comparison with the values at age of 3 days. In the case of AAS_SRA specimens, the decrease was not so significant – about 75% for modulus of elasticity and fracture toughness and about 60% for fracture energy. Contrary to these parameters, compressive strength values of both AAS and AAS_SRA specimens noticeably increased between 3rd and 28th day. Such evolution of mechanical properties can be explained by
microcracking since fracture parameters are more sensitive to any cracks compared to compressive strength. This is also supported by the results from length changes measurements where beneficial effect of SRA on AAS shrinkage was observed and the decrease in fracture parameters of SRA containing mortar between 3rd and 28th day is lower compared to the reference.

At the age of 90 days, the values of mechanical fracture parameters increased. In the case of AAS specimens, the increase was about 13, 33 and 67% for modulus of elasticity, fracture toughness and fracture energy, respectively, in comparison with values at the age of 3 days. In the case of AAS_SRA specimens, the values were 2–3 times higher than the values at the age of 28 days. In comparison with the values at the age of 3 days, only the fracture energy value increased by about 10%, the modulus of elasticity and fracture toughness values decreased by about 40 and 33%, respectively. Figure 5 shows the details of crack propagation from the initial notch for specimen after fracture tests at the age of 28 days (left) and at the age of 90 days (right). Obviously, the surface of the specimen is disturbed with the cracks, due to a high susceptibility of AAS composites to shrinkage. These cracks are visible at the age of 28 days, while no shrinkage-cracks are visible at the age of 90 days. Supposedly, self-healing occurred in the AAS composites, its origin will be a topic for further study. Note that all the test specimens were stored in the climate control chamber with the temperature of 21 ± 2°C and relative humidity of 60 ± 10% until their testing and were not protected from drying during whole time of ageing.

![Figure 5. Details of crack propagation from initial notch, specimens at the age of 28 (left) and 90 days.](image)

The influence of the specimens manufacturing process was assessed at the age of 90 days. The charts show that there are not significant differences between the results obtained from testing of the specimens casted into the moulds and specimens which were prepared by cutting from bigger specimens primarily used for length changes measurements. In most cases, the difference was about 10%.

The informative compressive strength values were determined on halves of specimens remaining after the fracture tests were performed. The compressive strength values gradually increased with age of specimens for both studied composites (see Figure 6), which is not in correspondence with obtained unexpected decrease of all investigated mechanical fracture parameters values at the age of 28 days.

The obtained mechanical fracture parameters are supplemented by the results of relative length changes measurements performed on both investigated composites. Figure 7 shows the values of relative length changes at the same ages when the fracture tests were performed. Note that in the case of AAS composite, the results are only for one specimen, because another two specimens cracked during the first days in the mould and it was not possible to continue the length changes measurements correctly. In the case of AAS_SRA composite, the mean values and standard deviations obtained from three independent measurements are introduced. The results show that the addition of SRA reduced the relative length changes by about 25% for all investigated ages of specimens. Details regarding the development of the length changes during the whole measurement can be found in [10].
Figure 6. The values of informative compressive strength of alkali-activated fine-grained composites.

Figure 7. The values of length changes of alkali-activated fine-grained composites.

5. Conclusions
The paper deals with the investigation of the values of mechanical fracture parameters of AAS composites. In particular, the effect of the shrinkage-reducing admixture (SRA) was studied. It was found that the addition of SRA reduced the relative length changes by about 25% at the age of 90 days. On the contrary, the addition of SRA had a negative impact on the values of the mechanical fracture parameters that is reflected in the decrease of values of about 50–60%. An exception was the results
obtained at the age of 28 days, when the monitored mechanical fracture parameters were higher in the case of AAS composite with SRA than in the case of AAS composite without SRA.

If the composites are evaluated separately and the trend of development of the fracture parameters is assessed, an unexpected decrease of all investigated parameters was observed for both tested AAS composites at the age of 28 days. It is suggested that more measurements have to be performed to confirm presented results and to clarify the reason for the decrease of monitored parameters values at the age of 28 days. Therefore, similar experiment will be designed and performed.

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