The effect of nano-additive TiO_2 on the failure process of self-compacting concrete assessed using the acoustic emission method

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Abstract. Due to the new challenges posed to engineering constructions, as well as the principles of sustainable development, many laboratories around the world are carrying out works to improve the basic structural material that is concrete. There has recently been a lot of interest in modifying concrete with nano-sized particles. Literature reviews indicate that their addition significantly improves the physical and mechanical properties of the concrete that was obtained with their use. Until now, there is no knowledge of the effect of nano-additives on the process of destroying temporarily compressed concrete. One test method that enables the parameters that describe the stress failure of concrete to be determined is the acoustic emission method. This work fills the gap in the literature and presents the results of the author's own research on the impact of the use of different amounts of nano-additive TiO_2 on the failure process of self-compacting concrete that was made solely on the basis of granite aggregate. The stress failure of the tested concrete was described using the stress levels (determined using the acoustic emission method) that initiate the cracking σ_i and critical stresses σ_{cr} that delimit the tested process. The descriptors that were used for this purpose are the rate of counts and the average effective value of the acoustic emission signal.

1 Introduction

Self-compacting concrete (SCC) is widely used in the construction industry. Compared to traditional concrete i.e. ordinary concrete, self-compacting concrete offers many advantages, among others: a lack of the need for mechanical compaction of the concrete mix, a low level of noise during concrete works, a lack of segregation effect of a concrete mix, the ability to tightly fill in a formwork, the exact coverage of a reinforcement, compaction under self-weight, a fast concreting process, high quality and durability of elements made of it, and also a less demand for physical work [1-3].

The stress failure of temporarily compressed concrete is an important issue from both the point of view of durability and the safety of constructions made of cement composites [4-6]. Based on research, it was found that three stages, which are qualitatively different in terms

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of damage to the concrete structure, can be identified in the concrete failure process. The individual stages are as follows: stable initiation of micro-effects created or initiated without the involvement of an external load, stable development of technological micro-defects and micro-cracks, and also unstable development and propagation of micro-cracks. The boundary thresholds at the transition between the individual stages are stress levels that are called the initiating cracking σ_i and critical σ_{cr} stress levels [7]. There is a relationship between the above-mentioned stress levels and the technological factors affecting the concrete. These factors are related to, among others, the composition of a concrete mix [8] and include the type of additive used in the concrete, which was shown in [9] using an example of fly ash and silica dust. It is proven that the level of stress that initiates cracking σ_i is identified with the fatigue strength of concrete, and the level of critical stresses σ_{cr} is related to the long-term compressive strength of concrete [7, 10].

One method of investigating the stress failure of compressed concrete is the acoustic emission method (EA). It is based on the phenomenon of the formation and propagation of elastic waves, which are due to the release of elastic energy accumulated in the material [11]. Acoustic emission transducers register elastic waves and transform them into electrical voltage. Afterwards, registered signals, after being processed in measuring apparatus, obtain the form of acoustic emission descriptors, such as the rate of acoustic emission counts, the number of acoustic emission counts, or the average effective value of the acoustic emission signal (RMS). The application of the acoustic emission method allows the behaviour of the material under the influence of a load to be continuously followed, and also the micro-changes in the structure and any increasing defects to be signalled [12].

The literature review shows that there were attempts in recent times to assess the effect of nano-additives on the physical and mechanical properties of the cement [13]. In the context of being a potential additive for the production of concrete, the following compounds are considered: SiO_2 , Al_2O_3 , CuO , TiO_2 , ZnO_2 , Fe_2O_3 and Cr_2O_3 [14]. As a result of their high chemical reactivity and the fact that they act as nuclei, around which the products of the cement hydration process grow, the nanoparticles are nano-filler of the concrete microstructure, which effectively reduce undesirable calcium hydroxide. They also create additional amounts of the C-S-H phase in the concrete structure [15]. Therefore, they improve the physical properties of concrete by reducing, among others, porosity [16] and permeability [17], as well as increasing the corrosion resistance of the concrete [18].

At the same time, the nanoparticles have a positive effect on the mechanical properties of concrete, increasing its micro-hardness [19] and compressive and bending strength [20]. TiO_2 nanoparticles, due to their photocatalytic properties, additionally counteract the accumulation of dust, dirt and fungi on the surface of concrete, and also purify the air [21]. The only disadvantage of nano- TiO_2 seems to be an economical aspect what was reported, among others in study [22], however, recently thanks to the new preparation methods like liquid phase deposition or sol-gel method [23] products like self-cleaning plasters containing TiO_2 nanoparticles appeared and they are commercially available at a reasonable price. Despite the advantages of modifying cement composites with nanoparticles, studies on the failure of concrete with nano-additives are highly selective [24].

Due to the increasing demands posed to structural materials, as well as the promising results of research on the modification of cement composites with nano-additives, it seems reasonable to study the effect of adding TiO_2 nanoparticles on the failure process of self-compacting concrete. The purpose of the study is to determine, using the acoustic emission method, the levels of stresses that initiate the cracking σ_i and critical stresses σ_{cr} in self-compacting concrete made using only granite aggregate with the addition of TiO_2 nanoparticles, which were then compared with the results for the reference concrete. The obtained results allow the existing gap in the literature to be filled and important information

with regards to the possibility of using nanomaterials as a potential additive for the production of concrete to be provided.

2 Test description

The self-compacting concrete mixes used for testing were made of the following components: Portland cement CEM I 52.5 R with a specific surface area of 0.6 m²/g and a density of 3.15 g/cm³, which was produced by Finnsementti Oy and met the requirements of EN 197-1 [25]; superplasticizer (SP) Glenium Sky 600 with a density of 1.06 g/cm³, which was applied in the amount of 4.0% of the cement weight; tap water and also granite aggregate with an average density of 2.67 g/cm³, fractions of 10-5, 5-2, 2-1, 1.2-0.5, 0.6-0.1 mm and a fraction with a grain size of <0.1 mm that acted as a fine filler. The grain curve of the used aggregate is shown in Figure 1. The W/C ratio of the designed mixes was equal to 0.42.

Fig. 1. The grain curve of the used granite aggregate

The composition of the concrete mix was modified with the nano-additive TiO₂ (Fig. 2) in an amount of 2.0% and 4.0% of the cement weight. Nanoparticles were in the form of a powder with a particle size of < 25 nm, a density of 3.9 g/mL at 25°C, a purity of 99.7% and a specific surface area of 50 m²/g.

Fig. 2. SEM image of the TiO₂ nanoparticles used in the tests.

From the above-listed ingredients, 3 mixes of self-compacting concrete were prepared. One mix was made without a nano-additive as a reference and the other two were modified with the addition of nano-TiO₂ in amounts of 2.0% and 4.0% of the cement weight. The summary of concrete mix compositions, calculated per 1 m³, is presented in Table 1.

Table 1. Compositions of the designed mixes of self-compacting concrete, calculated per 1m³.

Mix	Ingredients				
	Cement [kg]	Aggregate [kg]	Water [kg]	Nano-additive[kg]	SP [kg]
S1	460.0	1640.0	193.2	-	18.4
S6 (2.0% TiO ₂)	450.8			9.2	
S7 (4.0% TiO ₂)	441.6			18.4	

Homogenization of each mix was carried out in a mechanical mixer in two stages. The cement with aggregate with the addition of nanoparticles was first mixed for 2 minutes, and then after the addition of water combined with a superplasticizer, the mix was mixed for a further 2 minutes. From the aforementioned concrete mixes, 100 x 100 x 100 mm cubes were prepared, which were matured in a climatic chamber at an air temperature of 20°C (± 1°C) and relative air humidity of 95% (± 5%). After a period of one year, from the previously prepared cubes, four specimens with dimensions of 50 x 50 x 100 mm were cut out for the purpose of conducting tests using the acoustic emission method.

The tests using the acoustic emission method were made with the use of a Vallen-Systeme GmbH AMS3 apparatus set, two VS 150-M sensors with an available 100-450 kHz frequency band and also a strength machine. During the tests, the recorded acoustic emission descriptor as a function of time was the sum of counts and the effective value of the acoustic emission signal (RMS). During compression of the samples, in order to eliminate the friction at the interface between them and the pressure plates, the surfaces of the samples were grounded to an accuracy of 0.05 mm until they reached parallelism, and then a thin layer of technical grease was applied. The view of the acoustic emission test stand is shown in Figure 3.

Fig. 3. View of the test stand for the measurement of acoustic emission

3 Results of tests and their analysis

Figures 4, 5 and 6 show the averaged results of the rate of counts and the course of the effective value of the acoustic emission signal (RMS) that were registered in the function of the compression time of the tested concretes for series S1, S6 and S7. The diagrams also show an increase in the relative value of compressive stresses σ_c/f_c as a function of time of compressing specimens. Moreover, the averaged levels of stresses that initiate cracking σ_i and critical stresses σ_{cr} are indicated.

Fig. 4. The rate of the acoustic emission counts (a) and the effective value of the acoustic emission signal (RMS) (b) with the graph of the relative value of the increase of the compressive stresses σ_i/σ_c as a function of time of failure in the self-compacting concrete S1.

The presented research results confirm that the failure process of all the tested concretes had three stages. In the rate of counts and the effective value of the acoustic emission signal (RMS), two thresholds that distinguish different stages are noticeable. The first, less pronounced, corresponds to the level of stresses that initiate cracking σ_i , and the second, which is very distinct, corresponds to the level of critical stresses σ_{cr} . The obtained levels of

these stresses are different depending on the tested series. They amount respectively, for the reference concrete (S1) to $0.52 \sigma_c/f_c$ and to $0.78 \sigma_c/f_c$, for the concrete with the addition of 2.0% of nano-TiO₂ (S6) to $0.52 \sigma_c/f_c$ and to $0.82 \sigma_c/f_c$ and for the concrete with the addition of 4.0% of nano-TiO₂ (S7) to $0.68 \sigma_c/f_c$ and to $0.83 \sigma_c/f_c$. It is significant that the addition of TiO₂ nanoparticles in the amount of 4% results in a considerable increase in the levels of stresses that initiate cracking σ_i and critical stresses σ_{cr} . This may be the result of the improvement of the microporosity of concrete due to the addition of nano-sized particles, as was shown on the basis of studies of the same concrete in publications [26,27].

Fig. 5. The rate of the acoustic emission counts (a) and the effective value of the acoustic emission signal (RMS) (b) with the graph of the relative value of the increase of the compressive stresses σ_i/σ_c as a function of time of failure in the self-compacting concrete S6.

Fig. 6. The rate of the acoustic emission counts (a) and the effective value of the acoustic emission signal (RMS) (b) with the graph of the relative value of the increase of the compressive stresses σ_i/σ_c as a function of time of failure in the self-compacting concrete S7.

Additionally, in Table 2 set of results of initiate cracking stresses σ_i , critical stresses σ_{cr} and compressive strength f_c of conducted tests for each probe is shown. In turn, in Table 3, as a summary, average values of above mentioned parameters for each series are presented.

Table 2. Set of results of initiate cracking stresses σ_i , critical stresses σ_{cr} and compressive strength f_c for each probe

Series and probe	S1.1	S1.2	S1.3	S1.4-	S6.1	S6.2	S6.3	S6.4	S7.1	S7.2	S7.3	S7.4
σ_i [MPa]	20.6	20.4	19.1	18.5	20.7	20.9	20.1	20.3	26.8	25.7	25.8	26.1
σ_{cr} [MPa]	30.4	30.1	29.0	28.5	32.6	32.8	32.0	31.8	32.0	31.4	31.6	32.3
f_c [MPa]	39.2	38.8	37.2	36.0	40.0	40.2	38.6	38.8	39.0	38.6	38.0	37.7

Table 3. Average values of initiate cracking stresses σ_i , critical stresses σ_{cr} and compressive strength f_c for each series.

Series	S1	S6	S7
σ_i [MPa]	19.6	20.5	26.0
σ_{cr} [MPa]	29.5	32.3	31.8
f_c [MPa]	37.8	39.4	38.3

4 Summary

The paper presents the results of tests (using the acoustic emission method) of self-compacting concrete made solely on the basis of granite aggregate with the addition of TiO₂ nanoparticles used in various amounts. For each of the tested concretes, the course of the compressive stress failure was confirmed to have three stages. It was shown that the modification of the composition of concrete with the addition of TiO₂ nanoparticles increases the levels of stresses that initiate cracking σ_i and critical stresses σ_{cr} . This is probably the effect of, among others, the high reactivity of the additive, which is signalled in literature, and also the reduction of the porosity in the micro and nano pore range.

Considering the fact that the level of stresses that initiate cracking σ_i is considered to be equal to the fatigue strength of concrete, it can be concluded that the durability and safety of structures made of self-compacting concrete containing TiO₂ nanoparticles subjected to repeatedly variable loads will be higher in relation to a construction made of concrete without this additive. Similarly, an increase in the level of critical stresses σ_{cr} in concrete containing the addition of nano-TiO₂ can be interpreted favourably, assuming that this level is considered as the long-term compressive strength of concrete. On the other hand, concrete is destroyed more in a non-signalling way with an increase in the level of critical stresses σ_{cr} .

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