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# Evaluation of Shrinkage, Mass Changes and Fracture Properties of Fine-aggregate Cement-based Composites during Ageing

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**Abstract**

The paper deals with the experimental determination of relative length changes development during cement based composite ageing. Special moulds with the dimensions 100 × 60 × 1000 mm were used for the measurements. These moulds were filled and placed on an advanced weighing table that enables continuous recording of mass losses caused by free drying of the specimen surface. In this way, the mass losses and changes in the length of the cement composite in the moulds were measured simultaneously. The shrinkage moulds were modified in order to measure also the long-term relative deformation of fine-aggregate concrete specimens as a result of drying. The outputs of the measurement are given in the form of the diagrams displaying the relationship between the relative length changes or the mass losses and the time of cement composites ageing. Besides the measurement of length changes, three-point bending fracture tests at the age of 3 and 28 days were also performed. The modulus elasticity, effective fracture toughness and specific fracture energy values were determined from load versus deflection diagrams recorded during fracture experiments on the specimens with dimensions 40 × 40 × 160 mm.

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*Keywords:* cement composite; volume changes; shrinkage; mass loss; fracture test

## 1. Introduction

The determination of the detail development of volume changes of cement-based composites still remains in the focus of civil engineers and concrete producers. Along with the design of new materials, new factors influencing

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changes in volume appear throughout the whole time of their setting and hardening. Advancement in technology and composition of building materials in turn requires advancement in test procedures used for the determination of new materials' physical and mechanical parameters. The current approach in the field of material testing is aimed at identifying disruptions in the internal structure of structural elements as early as possible. This facilitates early diagnostics of the problem which allows for relevant precautions preventing later collapse of the building to be designed.

The reasons for the focus on this area are arising from the fact, that many problems with cement composites cracking are originated in the early ages [1,2,3]. Cracks formation and their propagations are for civil engineers closely associated with the tensile strength value, which is generally only 10 percent of compressive strength, and of course with the fracture parameter values. At early ages, the strength is still developing while stresses are generated by volume changes. Controlling the variables that affect volume change can minimize high stresses and cracking [2,4,5]. Experience gained from measurements performed in recent years suggests the necessity of assessing the magnitude of shrinkage in two consecutive stages of concrete ageing – in the early age and at later ages [5,6,7,8]. From measurements performed in the early-age cement-based composites, differences in the development of the volume changes can be identified, as well as differences in the initiation and propagation of cracks which cannot be identified with usual measurement started after removing the specimens from the moulds (typically after 24 hours) [9].

## 2. Experimental part

### *2.1. Materials*

For purpose of experimental measurement, three fine-grained cement composites mixtures were designed and manufactured. They differed in the water to cement ratio (*w*/*c*) and in amount of plasticizer. A design of composition is based on the standard ČSN EN 196-1 [10]. The fresh composite was made with quartzite sand with the maximum nominal grain size of 2 mm standardized according to ČSN EN 196-1 [10], Portland cement type 42.5 R and water in ratio of 3:1:0.5 (S:C:W), 3:1:0.47 and 3:1:0.35 with addition of super-plasticizer SVC 4035 in amount of 1 % by cement mass. A mixing device with controllable mixing speed was used to prepare the fresh mixtures. Together 15 specimens were prepared from each mixture (see Fig. 1). The basic information about the composition, manufacturing and properties of the fresh composite are given in Table 1. The properties of the fresh composite were determined in accordance with ČSN EN 1015-3 [11] and ČSN EN 1015-6 [12].

Table 1. Composition and properties of fresh composites.

| | | Composite ID | | |
|---|---|---|---|---|
| Components and properties | Units | 0_04042016 | III_02052016 | IV_09052016 |
| Sand | [kg] | 45.9 | 45.9 | 45.9 |
| Cement I 42.5 R | [kg] | 15.3 | 15.3 | 15.3 |
| Water | [kg] | 7.65 | 7.16 | 5.35 |
| Super-plasticizer SVC 4035 | % by cement mass | – | – | 1.0 |
| *w*/*c* ratio | [–] | 0.5 | 0.47 | 0.35 |
| Mixing speed | [revolutions per minute] | 20 | 30 | 40 |
| Workability | [mm] | 140.0 | 127.5 | 135.0 |
| Bulk density | [kg/m$^3$] | 2200 | 2210 | 2280 |

## *2.2. Test equipment and procedures*

### *2.2.1 Determination of volume changes progress*

The measurement procedure intended for determination of cement composites' volume changes was designed to record simultaneously the relative length changes, mass losses caused by free drying, temperature inside the tested specimen and ambient temperature and relative humidity. The measurement of shrinkage was performed using a test device made by the company Schleibinger Geräte Teubert u. Greim GmbH [13]. Special moulds of 1000 mm in length and with 60 × 100 mm in cross-section were used for recording the length changes measured along the central axis of the specimens using an inductivity sensor leaning against the movable head of the mould. These moulds are primarily designed for shrinkage measurement in the early stage of cement composites setting and hardening. Special markers were embedded into the upper surface of the cement composite placed in the shrinkage moulds in order to facilitate subsequent long-term measurement of relative deformation. In this way, two gauging bases were created for further measurement (see Fig. 1). This arrangement enabled the capture of the total relative length changes of the composite since the time the composite is placed into the mould until its long-term ageing after the specimen is removed from the shrinkage mould. Details about the markers types, drawing and their arrangement can be found in [14]. The polyethylene foam mat (MIRELON) of 2 mm thickness was placed on the bottom and along both longitudinal sides of shrinkage moulds in order to ensure free movement of the specimen in the mould. The shrinkage moulds were filled with fine-grained cement composite and placed onto a special weighing table that allowed continuous recording of mass losses caused by free drying of the specimen surfaces. For more details about the weighing table see [15].

The thermal sensor COMET was embedded at the end of shrinkage moulds in order to measure temperature inside the specimen. The ambient temperature and relative humidity were recorded by automatic gauging station COMET.

This measurement configuration enabled simultaneous measurement of all investigated parameters of the composite placed in shrinkage mould. The final arrangement of measurement devices before starting the measurement is shown in Fig. 1.

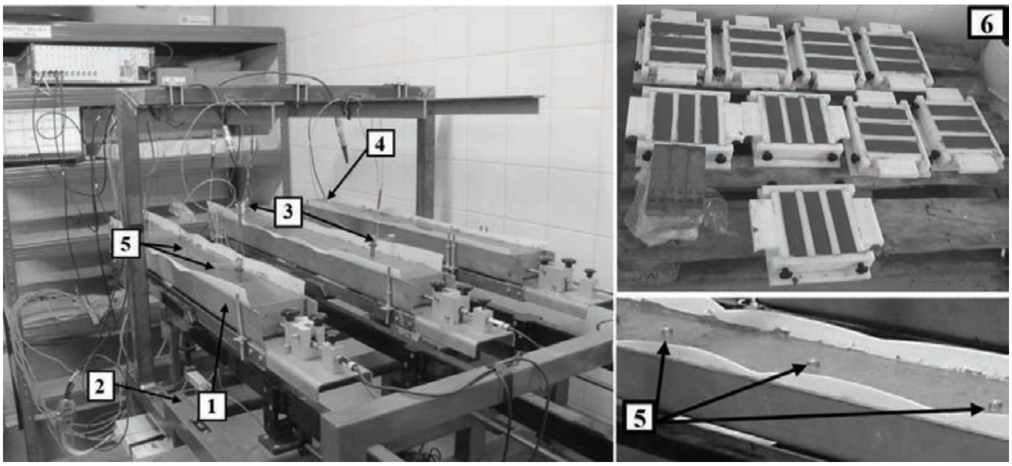


Fig. 1. Measurement equipment and test specimens (1 – shrinkage mould; 2 – weighing table; 3 – acoustic emission sensor; 4 – temperature probe; 5 – markers for long-term measurement; 6 – specimens for fracture tests).

It must be noted that the measurement in shrinkage moulds can only be started after the composite has set a little so that it does not push out the movable head of the mould with its own weight. Due to the consistency of the fresh composite, measurement was started approximately one hour after the composite was poured into the moulds. Length change was measured in the moulds placed on the weighing table in a laboratory at temperature of (21 ± 2) °C and relative humidity of (60 ± 10) % until the composite was 3 days old. The top surface of the composite was not protected from drying. The specimens were then extracted from the moulds, placed on the table and were exposed to free drying in a laboratory at a stable temperature of (21 ± 2) °C and relative humidity of (60 ± 10) %. Further measurements were performed using a Hollan's strain gauge [16] which was fixed onto the surface of the specimens. The positions of the gauging points were predefined by the markers embedded at spacing of 200 mm. The specimens were then left to dry freely for the entire time of the measurement and were weighed at regular intervals.

### *2.2.2 Fracture test*

Along with the volume changes measurements, the three-point bending fracture tests were carried out using a Heckert FP 10/1 testing machine with measuring range of 0–2000 N. Beam specimens with nominal dimensions of 40 × 40 × 160 mm and initial central edge notch were tested at the age 3 and 28 days. The displacement increment loading was performed, which allowed to record load versus displacement diagrams (*F–d* diagrams) during the tests. The *F–d* diagrams were used for the determination of elasticity modulus from the first (almost linear) part of the diagram, and for the calculation of effective fracture toughness using the effective crack extension method [17] and specific fracture energy using work-of-fracture method [18]. Because of stability loss during loading, it was not possible to reconstruct the descending part of *F–d* diagrams. Therefore, the work of fracture value is determined as area under *F–d* diagrams before stability loss occurred. For detail about determination above mentioned mechanical fracture parameters see [19].

## 3. Results and discussion

The results of performed measurement are presented in following figures and tables. Fig. 2 shows the progress of relative deformation and mass losses measured in the early stage of composite ageing. The data were obtained from measurements performed on the specimens in the shrinkage moulds placed on the weighing table. Apparently, different progress of shrinkage and mass losses was recorded for mixtures being investigated (see Fig. 2). Shortly after start of the measurement both mixtures without plasticizer exhibited swelling (length increment) of the test specimens, while rapid shrinkage of specimens with plasticizer was observed (see Fig. 2 left).

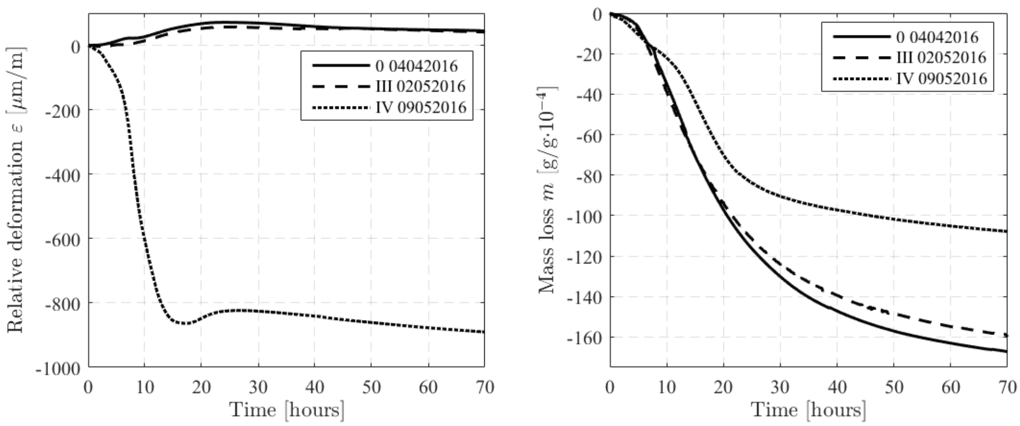


Fig. 2. Relative deformations and mass losses vs. time measured within first 72 hours of specimens ageing.

Also, the progress of mass losses recorded within first 72 hours of composites ageing is different for mixtures with and without plasticizer. The mass losses of both composites without plasticizer are higher than in the case of composite with plasticizer. This progress is closely linked with the adjustment of water content. Once the plasticizer is added, the water content is reduced which influences the progress of volume changes as well as mass losses. Apparently, the *w/c* ratio of the composites without plasticizer was rather high (see Tab. 1). No component segregation in the fresh mixture was observed either during the mixing or during the manufacturing of the test specimens. On the other hand, a relatively large amount of water rose to the upper surface of the test specimens after their manufacturing and storing.

This phenomenon is commonly known as bleeding [20]. Gradually, during the solidification of the composite, a part of the water evaporates from the top surface of the test specimens, causing mass losses of the specimens, and another part is drawn back into the pore structure of the composite, which contributes to the swelling of cement composite [7].

It is already known and has been proven in previous experimental measurements [21] that the addition of plasticizer has also a substantial influence on cement hydration. The addition of super-plasticizer retards cement hydration and has a critical influence on its overall progress. The margin of hydration retardation is strongly dependent on the amount and properties of the cement and the plasticizer used in the composite mixture [22].

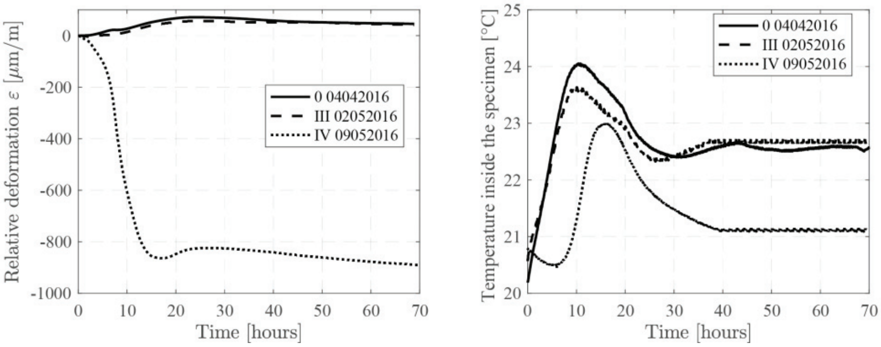


Fig. 3. Relative deformations and temperature measured inside the test specimen vs. time (within first 72 hours of specimens ageing).

The progress of shrinkage corresponds well with the progress of temperature measured inside the test specimens (see Fig. 3). In case of composites without the super-plasticizer, the highest temperature was recorded approx. 11 hours after start of measurement. At the same time, a surge of swelling (length increment) is also visible. This length increment is ascribed to the cement composites thermal expansion. Similar progress was recorded also for composite with the super-plasticizer. Presence of the super-plasticizer retarded early hydration and delayed temperature growth by approx. 5 hours (in comparison with composite without the plasticizer).

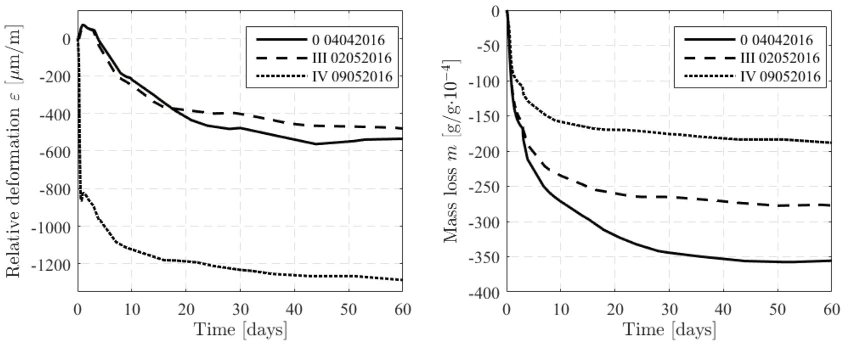


Fig. 4. Progress of relative deformations and mass losses measured over whole time of specimens ageing.

Concerning the results of long-term measurement, it can be stated that the addition of the super-plasticizer has a substantial influence on the total progress of relative deformations (see Fig. 4). The initial rapid growth of shrinkage influenced also its final values which are more than two times higher than the values recorded for cement composite without the super-plasticizer with *w/c* ratio of 0.5. The period of expansion (swelling) appears not to be very significant in terms of its magnitude. However, the initial expansion delayed the start of shrinkage by more than 20 hours. This delay can be of great benefit to the later development of physical and mechanical properties of cement composites. The total progress of mass losses corresponds well with the water content used in mixtures (see Table 1). The lowest mass loss was recorded for composite with the super-plasticizer with *w/c* ratio of 0.35.

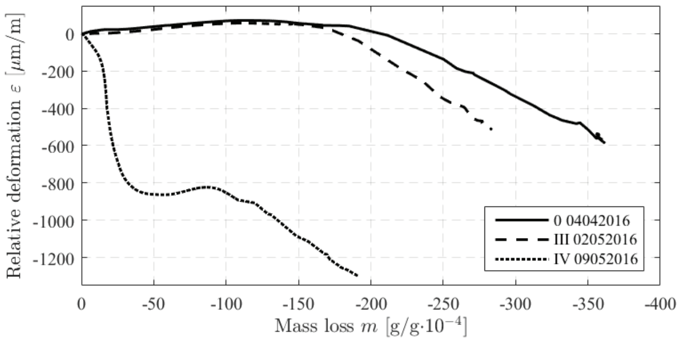


Fig. 5. Relationship between deformations and mass losses measured over whole time of specimens ageing.

The correlation between relative length changes and mass losses was observed over whole time of cement composite ageing (see Fig. 5). However, the initial part of the curve is influenced by water reabsorption and thermal expansion.

The box plots of selected mechanical fracture parameters at the age of 3 and 28 days, i.e. modulus of elasticity, effective fracture toughness and specific fracture energy, are introduced in Figures 6–8. Relative values of these parameters are summarised in Tables 2 and 3. Note that statistical differences between values of obtained parameters were tested by one-way analysis of variance (ANOVA) and one-way Kruskal-Wallis test. These methods were used for testing whether samples originate from the same probability distribution. Based on this analysis, differences between all composites are statistically significant with the significance level of 0.05 (–).

Table 2. Relative mean values (%) of mechanical fracture parameters: 100% = results at age of 3 days.

| | Composite ID / Age of specimens | | | | | |
|---|---|---|---|---|---|---|
| | 0_04042016 | | III_02052016 | | IV_09052016 | |
| | 3 days | 28 days | 3 days | 28 days | 3 days | 28 days |
| Static modulus of elasticity | 100 | 89 | 100 | 103 | 100 | 99 |
| Effective fracture toughness | 100 | 123 | 100 | 122 | 100 | 108 |
| Specific fracture energy | 100 | 168 | 100 | 142 | 100 | 121 |

Table 3. Relative mean values (%) of mechanical fracture parameters: 100% = results for composite ID 0_04042016 (*w/c* = 0.50).

| | Age of specimens / Composite ID | | | | | |
|---|---|---|---|---|---|---|
| | 3 days | | | 28 days | | |
| | 0_04042016 | III_02052016 | IV_09052016 | 0_04042016 | III_02052016 | IV_09052016 |
| Static modulus of elasticity | 100 | 89 | 115 | 100 | 103 | 127 |
| Effective fracture toughness | 100 | 98 | 140 | 100 | 97 | 122 |
| Specific fracture energy | 100 | 110 | 168 | 100 | 94 | 121 |

Values of mechanical fracture properties increased with age of almost all composites up to 10–70 %; exception showed values of elasticity modulus – a decrease more than 10 % was measured in case of composite 0_04042016, i.e. with the highest *w/c* ratio. Mechanical fracture parameter values of composites with similar *w/c* ratio were also very close for both ages of specimens – maximal difference was around 10 % for specimens at age of 3 days. These values significantly increased with decreasing of *w/c* ratio – in case of elasticity modulus it was up to 15 and 27 % for 3 and 28 age, in case of fracture toughness there was an increase up to 40 and 22 %, and in case of fracture energy it was up to 68 and 21 %.

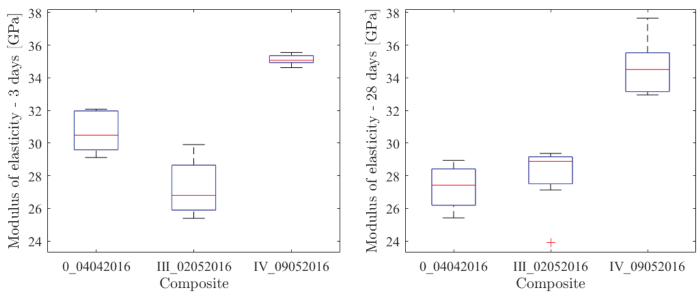


Fig. 6. Modulus of elasticity (outlier – the cross in the graph).

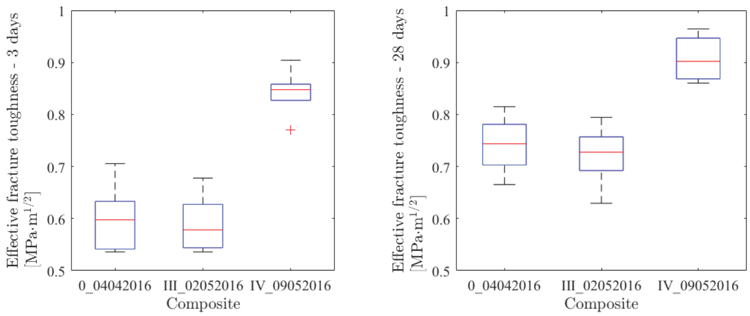


Fig. 7. Effective fracture toughness (outlier – the cross in the graph).

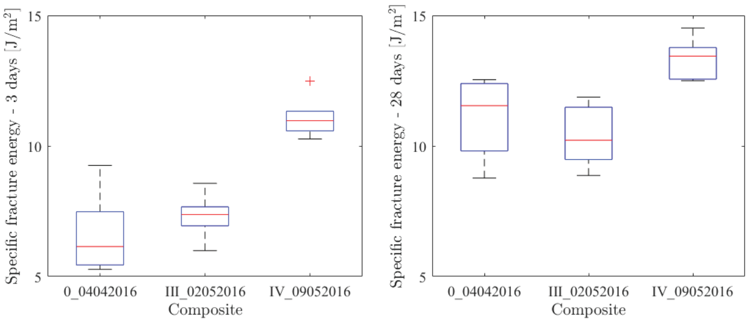


Fig. 8. Specific fracture energy (outlier – the cross in the graph).

## 4. Conclusion

The measurement technique described in the paper and used in the experiment satisfies the requirements for the early age diagnosis of the material's behaviour very well. The measurement of relative length changes together with the recorded progress of mass losses and temperature measured inside the test specimen provided comprehensive information about the behaviour of material in the early stage of its setting and hardening. The possibility of continuation of the relative length changes measurement as well as mass losses measurement on the test specimens, after they were removed from the moulds, allowed obtaining the continual progress of investigated characteristics during the whole time of cement-based composites setting and hardening. Data obtained from the experiment are useful for creation the new and verification of the existing computational models designed for prediction of progress of volume changes in cement-based composites. Investigation and determination of mechanical fracture parameter values during the composite material ageing is of great contribution in case of description of the material resistance to the cracks formation and propagation. Creation of the relationship between the progress of relative length changes and development of mechanical fracture parameter values will be of great use in case of prediction of material behaviour throughout the whole time of its solidification.

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