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6. Comprehensive Monitoring of the Shrinkage and Structural Changes of Cement Composites during Setting and Hardening

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6.1. Introduction

Determination of the real shrinkage development of cement-based composites is ever more important not only for structural engineers and concrete manufacturers but it is also a very topical problem in the field of the development of mathematical models for the creep and shrinkage prediction in concrete structures (RILEM TC 2015). 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 main problem in the field of experimental measurement of shrinkage in cementitious materials is to capture the total volume change due to chemical reactions, plastic settlement and desiccation while recording all the characteristics that more or less influence the process and magnitude of shrinkage in particular stages of their setting and hardening.

The main aim of performed experiment was to find measurement equipment and test procedure suitable for obtaining comprehensive information about the structural and volume changes which are in progress especially during the early stage of cement composites setting and hardening.

6.2. Theoretical background

Immediately, after cement is mixed with water, structural and volumetric changes in fresh mixture are in progress (Bentz 2008, Newman & Choo 2003, Neville 2011, Bella et al. 2016). These changes are caused by many factors related to the environmental and curing conditions (Havlásek 2014) as well as to the properties and ratio of raw materials used in the fresh mixture (ed. by Kovler et al. 2006). One of the most monitored phenomenon, which is closely connected with above mentioned matters, is early-age cracking (ed. by Bentur 2003, Deshpande et al. 2007). The high risk of early cracks initiation and propagation occur especially in restrained (Ba et al. 2008, Dong et al. 2014, Loser & Leemann 2009) and massive structures (Lin et al. 2012, Nehdi et al. 2014) because of stresses developed by volumetric changes due to the chemical reactions, temperature development or drying. One of the current approaches to determination of relationship between structural and volumetric changes is the simultaneous measurement of relative length changes and acoustic responses due to chemical reactions, particles solidification or cracking. In recent years, the method of acoustic emission (AE) has also been widely used as a supplemental measurement method for the non-destructive monitoring of the changes in the specimen’s internal structure during static and dynamic loading tests as well as for the monitoring of the behaviour of composite materials during setting and hardening (Topolář & Pazdera 2014, Topolář et al. 2016), (Qin et al. 2014, Pirskawetz et al. 2006). The previous studies shown that the AE technique is able to detect the early-age crack formation and cracking progress during the cementitious composite solidification (Qin et al. 2014, Kim & Weiss 2003).

Scientific sources provide a number of approaches to determining the value of particular types of concrete and mortars volume changes (shrinkage, swelling), e.g. (Reinhardt & Grosse 2005, PCA 2017, Holt 2001), (Kratochvíl et al. 2014, Barcelo et al. 2005 Soliman & Nehdi 2011, Mazzoli et al. 2015, Amin et al. 2010, Zhang et al. 2015, Chen et al. 2011, Chen et al. 2010, Yılmaztürk et al. 2004). However, these are mostly methods for separate determination of individual components of shrinkage in the early age. In these cases, the measurement is started immediately or very early after cement is mixed with water. Measurement methods intended to the long-term monitoring of the shrinkage progress due to drying are mostly based on the determination of the relative length changes and the measurement, in most cases, begins after specimens have been removed from their moulds, which is typically no sooner than after 24 hours of ageing. Such methods are summarized e.g. in (Newlands et al. 2008) or they are standardized in the national standards of various countries.

In important or complicated concrete structures, shrinkage is measured directly on a concrete element using a special type of wire strain gauge designed to be embedded in the concrete (Norisham et al. 2008, ed. By Tanabe et al. 2009, Stráský et al. 2014, Zich 2011) or other advanced monitoring technique (Ma et al. 2015).

Guidelines reflecting the recent advances in theoretical and experimental research in the field of the creep and shrinkage of cement composites (especially concrete) have been published under RILEM TC-242-MDC (chair Zdeněk P. Bažant). An article from 2015 (RILEM TC 2015) underlines the necessity of measuring weight losses, especially at the early stage of cement composite ageing, because this characteristic is an important input parameter for the development of mathematical models which are used for the prediction of concrete volume changes. The uncertainty involved in extrapolating drying shrinkage can be considerably reduced in case the weight loss is measured simultaneously with shrinkage (RILEM TC 2015).

6.3. Materials and methods

Testing techniques

In order to select measurement equipment and test procedure suitable for obtaining the relevant measurement outputs, several specifications, which were based on the experience gained over the last ten years in the field of concrete volume changes measurement, were taken into account. The main emphasis was posed on the accuracy, ability to setup the continual measurement with the adjustable storage frequency, ability of continuous measurement of all investigated parameters without specimens handling and ability of simultaneous measurement of all investigated parameters such as length changes, mass losses caused by free drying, temperature development inside the test specimen, acoustic responses of internal structure changes and ambient temperature and relative humidity.

Based on the above-mentioned requirements following measurement equipment was selected. The measurement of length changes was performed using the u-shaped stainless steel shrinkage drains of 1000 mm in length and with 60 × 100 mm in cross-section (Schleibinger Testing Systems 2017) were used. The gauging bases are defined by two anchors placed on heads of drains. One anchor is fixed and the other one is movable and sliding on three wheels. To avoid wall friction the drains were covered with polyethylene foam mat (MIRELON) of 2 mm thickness. The length changes were measured along the central axis of the specimens using an Inductive Standard Displacement Transducers WA2T (HBM 2017) with measuring range of 2 mm. The greatest linearity deviation of these sensors is 0.2% of measuring range. The sensors leaning against the movable anchor of the drain were mounted to the drain in desired position.

This measurement equipment is primarily designed for shrinkage measurement in the early stage of cement composites setting and hardening. In order to facilitate the subsequent long-term measurement of deformations special markers (Kucharczyková et al. 2011) were designed at the Brno University of Technology (BUT). These markers were embedded into the upper surface of the composite placed in the shrinkage drains (see Fig. 6.1.). In this way, two gauging bases were created for further measurement (see Fig. 6.1., Fig. 6.2.). This arrangement enabled the capture of the total relative length changes of the concrete since the moment the concrete is placed into the drain until its long-term ageing after the specimen is removed from the shrinkage drain.

To provide the continuous measurement of mass losses a special weighing table (Vymazal et al. 2015) was also designed at the Brno University of Technology (BUT). The measurement accuracy is guaranteed by a rigid frame and high sensitivity single point load cells of the accuracy class C3MR (HBM 2017). The max. weight capacity is 3kg. The construction and measurement details are under national patent protection (Vymazal et al. 2015).

The shrinkage drains filled with the fresh mixture were placed onto a special weighing table which enabled the simultaneous measurement length changes and mass losses of the test specimens. Measurement data obtained from drains and weighing table were automatically stored using a universal amplifier Quantum X - MX 840 (HBM 2017) with 8 individually configurable inputs (electrically isolated). The data storage frequency was 5Hz. The setup of particular measurement devices before the start of measurement and data storage frequency was performed using the PC software CatmenEasy.

In order to measure temperature development inside the test specimens the platinum thermal sensor COMET PT 1000 was embedded at the end of the shrinkage drains. Measured data were storage with a period of 5 min using a thermometer-logger S0151 for 4 external probes (COMET 2017).

Changes in the specimens' internal structure during the early stage of setting and hardening were monitored using the non-destructive passive method of AE. Magnetic AE sensors (type MDK13 with 35 dB preamplifier) were placed in positions predefined by embedded steel probes (waveguides), see Fig. 2 and Fig. 3, which provide the transfer of acoustic waves generated during material's setting and hardening to the AE sensor.

The internal structure changes were monitored with two AE sensors placed in each shrinkage drain during the measurement – the first one was placed near the movable head of the drain, the second one was located near the other end of the drain. In this way, the progress of events during cement composites setting and hardening was continuously recorded. AE signals were detected by the measuring equipment DAKEL XEDO with two channels for approx. 72 hours. Universal measurement and diagnostic system DAKEL XEDO was used for measurement. This equipment allows sampling of the signal from the AE sensors (speed up to 8 MSamples/sec), enumerates standard acoustic emission parameters, process emission events parameters to localize the feasible (potential) emission source.

The measurement of AE was started simultaneously with the start of relative length changes, mass losses and temperature measurement. In order to evaluate the origin of microcracks during setting and hardening, the measurement was focused on the number of overshoots which exceed the pre-set threshold, which is the most commonly used parameter of the acoustic emission activity. The presence of a high number of microcracks in the specimen is reflected in the high acoustic emission activity.

The ambient temperature and relative humidity were continuously recorded with a period of 15 min by an automatic gauging station COMET (COMET 2017). The above-described measurement equipment and configuration enabled simultaneous measurement of all the parameters being investigated.

The final arrangement of the measurement devices before starting the measurement is shown in Fig. 6.1.

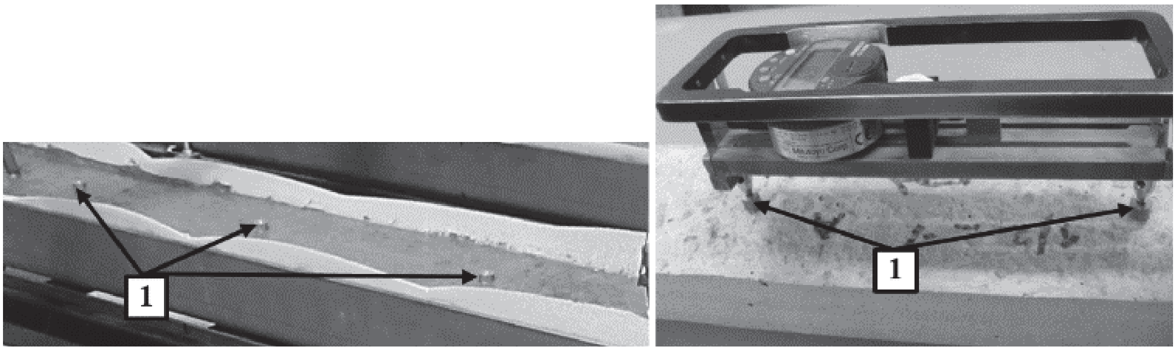


Fig. 6.1. Arrangement for long-term measurement using Hollan's strain gauge (1 – markers for long-term measurement)

The measurement in shrinkage drains can only be started after the concrete has set a little so that it does not push out the movable head of the drain with its own weight. With respect to the consistency of the fresh mixture, measurement was started approximately one hour after the composite was poured into the drains. Investigated parameters was measured in the drains placed on the weighing table in a laboratory at temperature of 21 ± 2 °C and relative humidity of 60 ± 10 % until the specimens was approx. 3 days old.

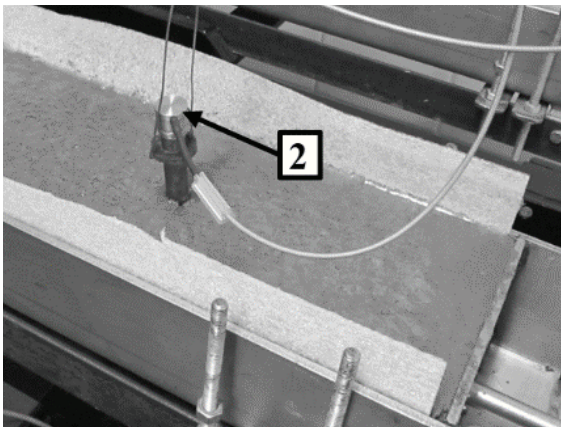


Fig. 6.2. AE sensor with waveguide in shrinkage mould

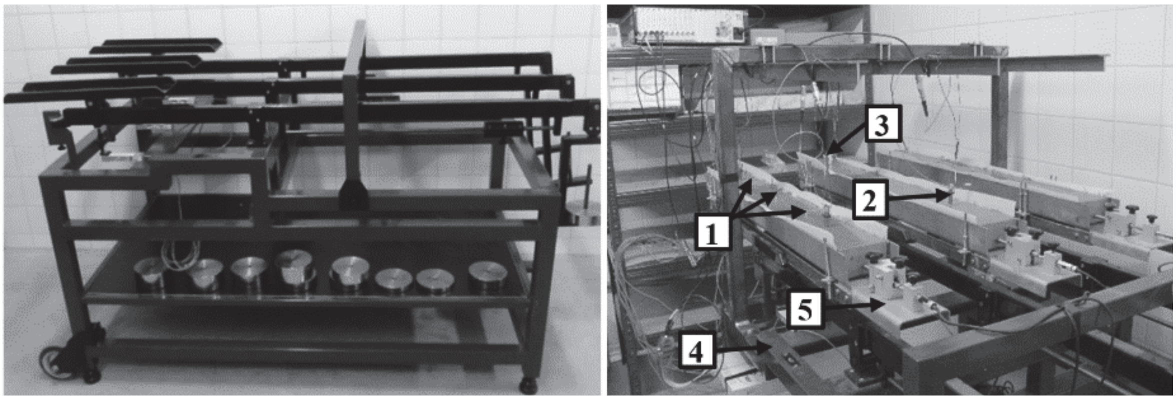


Fig. 6.3. Arrangement of measurement devices (1 – markers for long-term measurement; 2 – AE sensor with waveguide; 3 – temperature sensor; 4 – weighing table; 5 – shrinkage drain)

To verify the sensitivity and accuracy of the weighing table the top surface of the test specimens was not protected from drying. After 3 days the specimens were extracted from the drains and stored 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 which was fixed onto the surface of the specimens (see Fig. 1). The positions of the gauging points were predefined by the markers embedded at a spacing of 200 mm. The specimens were left to dry freely for the entire time of the measurement and were weighed at regular intervals.

Materials

The main aim of the experimental part was to verify the setup of the test equipment and test procedure intended for the determination of the volume changes in cementitious composites, especially in their early stage of setting and hardening. For this purpose, a fine-grained cement composite with a relatively high w/c ratio was designed and manufactured. The composition is based on the standard ČSN EN 196-1 (Part 1 2005). The fresh composite was made with quartzite sand with the maximum nominal grain size of 2 mm standardized according to ČSN EN 196-1 (Part 1 2005), Portland cement CEM I 42.5 R and water in ratio of 3:1:0.5 (S:C:W). A mixing device with controllable mixing speed was used for the preparation of the fresh mixture. The basic information about the composition, manufacturing and properties of the fresh composite are given in Table 6.1. The properties of the fresh composite were determined in accordance with ČSN EN 1015-3 (Part 3 2000) and ČSN EN 1015-6 (Part 6 1999). The basic properties of hardened composite are summarized in Table 6.2.

Table. 6.1. Properties of fresh composite

Composite ID	Composition [S:W:C]	w/c [-]	Mixing speed [revolutions/min]	Workability [mm]	Bulk density [kg/m^3]
0_04042016	3:1:0.5	0.5	20	140	2200

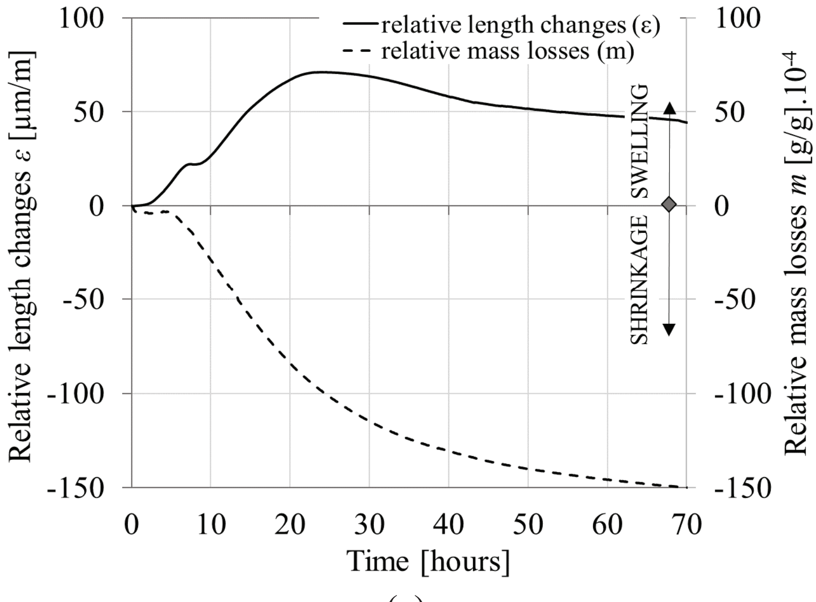
Table. 6.2. Properties of hardened composite

Composite ID	Age of specimens [days]					
	3	28	90	3	28	90
	Compressive strength [N/mm2] (standard deviation)			Dynamic modulus of elasticity [N/mm2] (standard deviation)		
0_04042016	27.28 (1.84)	36.53 (2.57)	37.62 (2.32)	27340 (669)	27735 (724)	28693 (492)

Note: For the purpose of performed experiment, the test specimens were intentionally no protected from drying during the whole time of ageing. This inappropriate way of curing probably led to decrease in the mechanical properties of the material being investigated.

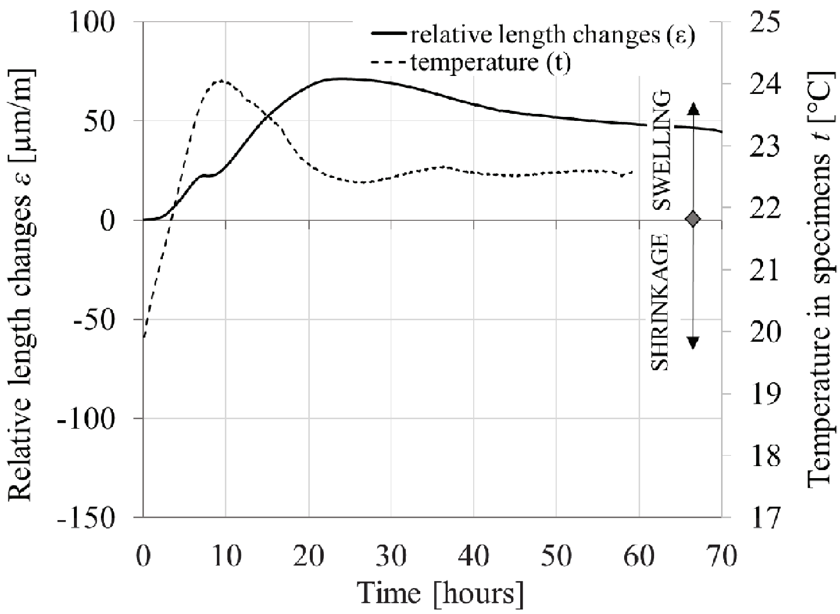
6.4. Results and discussion

The results of the measurements performed are presented below. Fig. 6.4. shows the progress of relative length changes, relative mass losses and temperature inside the test specimens measured in the early stage of setting and hardening. This method of displaying the recorded data provides a better idea about the relations between the phenomena being investigated. Shortly after the start of the measurement, swelling (length increment, expansion) of the test specimens was recorded. However, decrease in mass of the test specimens was recorded during the same time period as well. An explanation of this phenomenon can be found in the composition of the cement composite and in the development of the temperature measured inside the test specimens. Looking on the material composition it can be stated that the w/c ratio of this composite was rather high (see Tab. 6.1.). 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. After the plastic settlement has finished, the water which had bled onto the specimen upper surface is drawn back into the setting and later hardening cement paste, re-filling the pores created during cement hydration. This water re-absorption is one of the factors which cause the swelling of concrete (Holt 2001). Another substantial factor which affects the early swelling of cementitious composites is the thermal expansion due to the heat generated during hydration. The phenomenon of swelling/expansion is clearly visible from the measurements in the first 24 hours of ageing when the hydration heat is generated at the highest rate (approx. 200 J/g per 24 h) (Rovnaníková & Žalud 2015). The progress of temperature measured in the specimens confirmed this (see Fig. 4). The highest temperature was recorded approx. 10 hours after the start of measurement. At the same time, a surge of swelling is also visible. Its highest magnitude of 75 μm/m was recorded after 22 hours counted from the start of measurement. Concerning the recorded progress of mass losses, it is clearly visible that the sharp loss in specimens' mass was recorded just before reaching the maximum temperature.

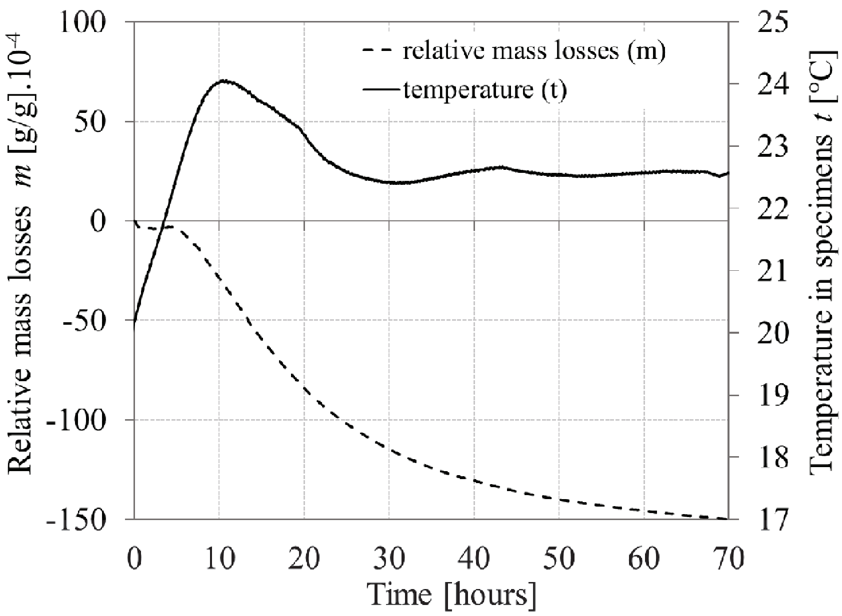


(a)

Fig. 6.4. Progress of relative deformations, relative mass losses and temperature measured inside the test specimens



(b)

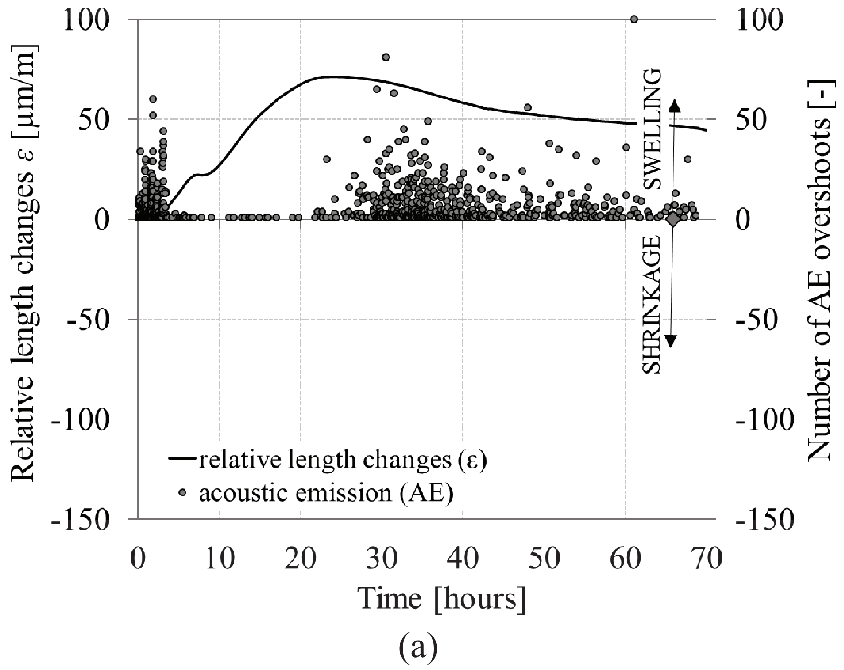


(c)

Fig. 6.4. cont. Progress of relative deformations, relative mass losses and temperature measured inside the test specimens

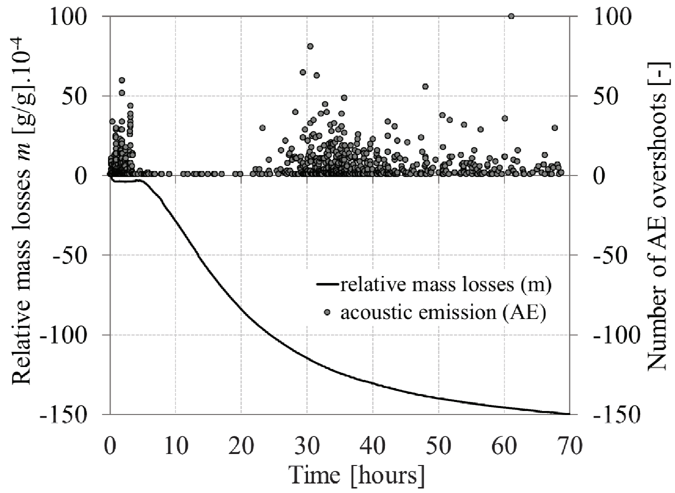
Fig. 6.5. shows the progress of relative length changes and mass losses in relation with the AE events obtained during the monitoring of the changes in the specimens' internal structure during the early stage of setting and hardening by means of AE measurement. The acoustic waves recorded by measurement

equipment were conversed to the number of overshoots for the purpose of results interpretation. This number of overshoots indicates the number and size of structural changes being in progress within a certain period. The more activity there is in the specimens' internal structure, the more overshoots are recorded per an interval of time. Fig. 6.5.(a) clearly shows that the activity recorded by the AE corresponded well with the progress of the composite relative length changes. The first high number of overshoots was recorded immediately after the start of the measurement. In this early stage of composite ageing, plastic settlement takes place, which is reflected in the high numbers of AE overshoots. After plastic shrinkage had finished, a period of swelling started. During the whole period of swelling only very low activity was recorded. The numbers of overshoot started to increase immediately after the swelling had stopped and the composite started to shrink (see Fig. 6.5.(a)). Based on the results, it can be assumed that no important internal changes are occurring during the stage of swelling. It means that probably neither water re-absorption nor thermal expansion are the initiators of cracks, in this case, and the composite gains strength without internal cracking. It should be emphasized that in this case the shape of specimen, especially its cross section, plays an essential role.

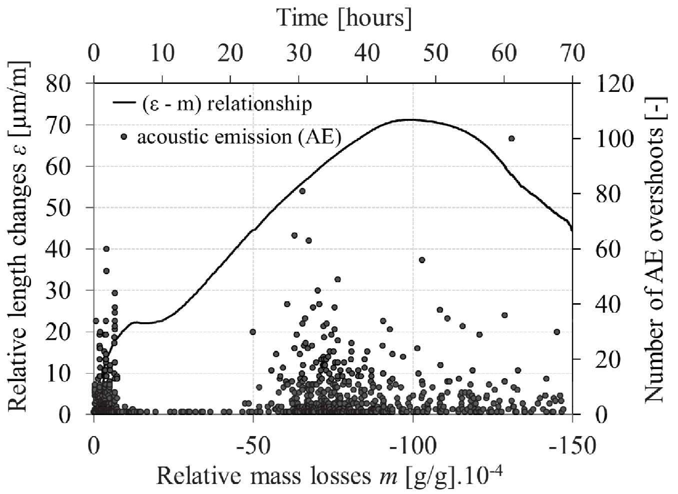


(a)

Fig. 6.5. Progress of relative deformations, mass loss and AE overshoots



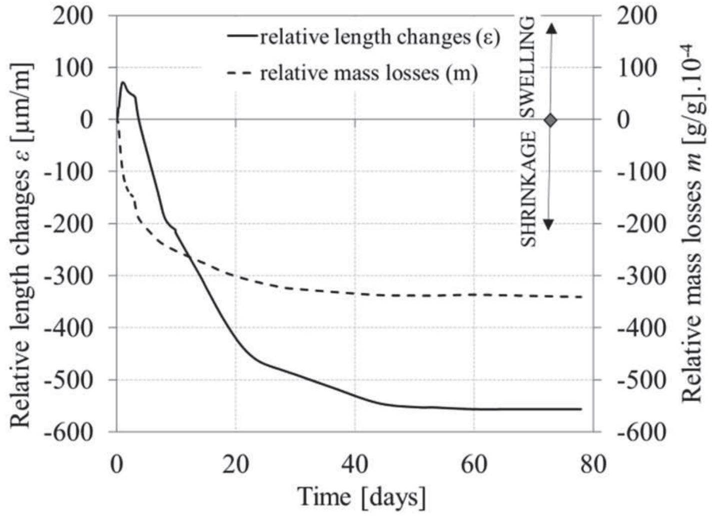
(b)



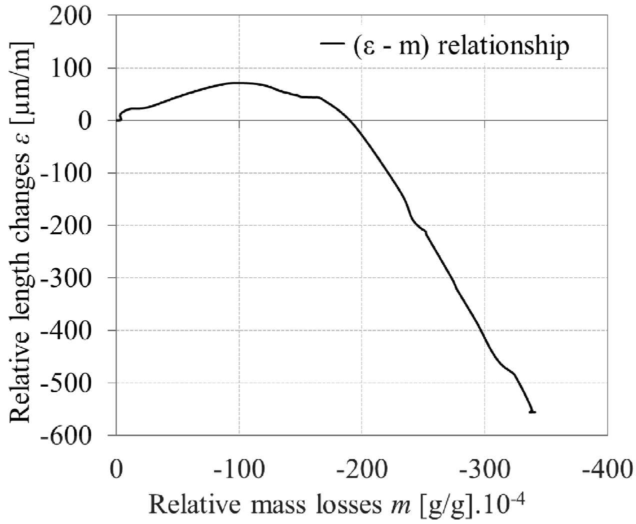
(c)

Fig. 6.5. cont. Progress of relative deformations, mass loss and AE overshoots

Concerning the progress of total volume changes, the period of initial expansion (swelling) appears not to be very significant in terms of its magnitude (see Fig. 6.6.(a)). However, from the perspective of strength development this phenomenon seems fairly interesting. Early age volume changes can influence tensile stress and crack formation in a hardened cement composite which is then visibly reflected in later development of its tensile and compressive strength as well as in the resistance of the composite to later crack formation and propagation. The period of swelling occurs within the first 24 hours when the cement composite is very delicate and the risk of internal cracking is very high. As stated above, the results of the measurements presented in Fig. 6.5. show a very low activity in the internal structure of hardened composite during the period of swelling – it can be assumed that no significant cracks are created. 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 the cement composite. The absence of early cracking in this period may result in overall increase in durability of the cement composite element.



(a)



(b)

Fig. 6.6. Progress of relative deformations and mass losses measured over the whole time of the composite's ageing

6.4 Conclusions

The measurement technique described in this chapter and used in the experiment satisfies the requirements for the early age diagnosis of the material's behaviour. Readings obtained from the shrinkage drains, weighing table, apparatus for detection of AE and devices for temperature and humidity measurement provided comprehensive information about the behaviour of the material especially in the early stage of its setting and hardening.

In conclusion, the measurement results suggest the following:

- Measurements have to start as soon as the cement composite has been poured into a mould if a comprehensive assessment of the parameters influencing volume changes is to be made. The consistency of the fresh mixture as well as the capabilities and limitations of the measurement equipment must be taken into account. The shrinkage drains can be considered suitable for the measurement of volume changes in cement composites of varied workability. The measurement should usually be started no later than one hour after the composite is poured into the drain. Generally speaking, the lower the workability of the composite, the sooner the measurement can begin.
- Continuous measurement of the mass losses of the cement composite caused by free drying of the specimen surface provided useful data for result interpretation of the measurement of volume changes in cement composites. This data then be of great use in creating new mathematical models designed for the prediction of volume changes in cement composites as well as in fine-tuning existing ones. The measured progress of mass losses corresponds well to the progress of relative length changes and to the progress of temperature measured inside the test specimens. The relationship between relative length changes and relative mass losses was observed over the whole time of its ageing (see Fig. 6.5.(b) and Fig. 6.6.(b)). However, the initial part of the curve is influenced by water re-absorption and thermal expansion. Just before reaching the maximum temperature a sharp loss in specimen mass was recorded (see Fig. 6.4.(c)). The measurement results can provide information as to how much the water loss and release of hydration heat contribute to the overall progress of volume changes.
- The method of acoustic emission appears to be another suitable tool for determining the structural changes and occurrence of micro-cracks during the setting and hardening of a cement composite. In general, the higher the numbers structural changes and micro cracks, the greater a number of AE overshoots is recorded. The number of micro-cracks in the cement composite essentially affects its final mechanical properties (i.e. strength, modulus of elasticity, fracture parameters, durability, etc.). The application of the AE method for continual monitoring of cement composites during the setting and hardening can be instrumental in the early detection of micro disruptions. This information about the behaviour of the material may be later used for improving the material composition or adjusting the curing method which

csimonovan both lead to designing cement composite structures with better properties and higher durability.

- The testing technique described herein is currently commonly used at the Institute of Building Testing of BUT, FCE. The measurement results were published in following papers (Kucharczyková et al. 2017, Topolář 2017)

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