Non Destructive Online Evaluation of Concrete Hardening Using Acoustic Emission and Harmonic Wave Spectroscopy

Koen VAN DEN ABEELE, Wendy DESADELEER, K.U.Leuven Campus Kortrijk, Kortrijk, Belgium
Geert DE SCHUTTER, Ghent University, Laboratorium Magnel for concrete Research, Gent, Belgium
Martine WEVERS, K.U.Leuven, Dept. of Materials Engineering, Leuven, Belgium

Abstract. The early stages in the hydration process of concrete are crucial in the development of its mechanical properties and its durability. We have monitored the first three days of the hydration process using an integrated system that combines Acoustic Emission (AE) analysis with Nonlinear Elastic Wave Spectroscopy (NEWS) techniques. With the AE technique, we realize a passive interrogation of sound generation in the concrete that is capable of online monitoring the internal microstructural activity. The nonlinear wave propagation technique is an active interrogation that probes the nonlinear elastic properties of the microstructure through the analyses of the second harmonic generation from a continuous wave transmission through the concrete sample. In addition, common parameters such as temperature and P and S wave speeds are also measured during the curing process at regular time intervals. The evolution in the AE events, and the linear and nonlinear behavior of concrete is analyzed as a function of the degree of hydration and interpreted for various initial compositions, in terms of the phase changes due to chemical reactions and mechanical setting.

Introduction

The hardening of concrete is a quite complex, but highly important process which significantly determines the concrete's overall and long term mechanical properties, such as the tensile strength, the bending stiffness and failure modes. During the early hydration stages an interfacial transition zone is created between the aggregates and the cement paste, with a thickness of up to 50 µm. A precise knowledge of the micromechanical properties of this zone during the successive phases of the hydration process is crucial in providing information on the concrete resistance, because primary defects in the interfacial layer form a preferred path for crack propagation and transport of aggressive agents threatening its durability.

Several non-destructive techniques have been developed and applied in that respect. Common ultrasonic reflection and transmission techniques were suggested and performed by Sayers and Dahlin [1], Boumiz [2-3] and Morin [4] to monitor the linear elastic material properties of young concrete by means of longitudinal and transverse wave velocity measurements. In combination with volumetric shrinkage measurements, they were able to interpret the evolution of the capillary network of a High Performance Concrete during hardening. In general, however, lack of sensitivity and difficulties in interpretation of the linear ultrasonic measurements arise when validating the early stages of curing [1-4]. In addition, the instantaneous structural activity in concrete at early ages has been monitored by passive ultrasonic means using Acoustic Emission (AE) Monitoring [5]. Even though the results show distinct periods of intense microstructural changes during the curing process, till now, the correlation between the number of AE-events and the micromechanical properties remains unclear, and a quantification of microstuctural transformations (i.e. chemical and physical alterations and/or damage) induced by chemical reactions and setting during curing is not easily achieved.

Alternatively, the particular aspect of monitoring the influence of induced porosity on the micromechanical properties of concrete during hardening, may well be handled by nonlinear dynamic mechanical measurements. Micromechanical features at the constitutive level, i.e. microscopic nonlinear stress-strain relations, result in ultrasonic wave distortion, which gives rise to changes in the resonance frequencies as a function of drive amplitude, generation of accompanying harmonics, nonlinear attenuation, and multiplication of waves of different frequencies [6-10]. Techniques exploring these signatures are commonly called Nonlinear Elastic Wave Spectroscopy (or short NEWS) techniques. In a series of laboratory NDT applications on various materials [10-12], NEWS techniques were found to be much more sensitive to mechanically and chemically induced structural alterations than any other method based on the investigation of linear material parameters such as wavespeed and damping.

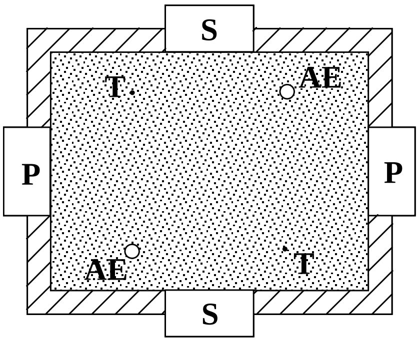
In order to simultaneously probe the instantaneous microstructural activity and the micromechanical characteristics of freshly poured concrete during its curing process, we developed an integrated system of dynamic non-destructive techniques based on AE (passive interrogation), linear ultrasonic wave propagation and NEWS harmonic monitoring (active interrogation). With this study, we complement and extend the work of Lacouture et al. [13] who started the use of NEWS for monitoring the first chemical reaction phase in the curing process. Using appropriate theoretical hydration models [14-15], we interpret these results within the framework of the degree of hydration concept. This should eventually lead to an improved prediction of the long-term behaviour of concrete and its performance dependence on the curing processes.

1. Concrete composition and integrated monitoring system

The monitoring experiments were performed on a cubicle measuring 200×150×100 mm^3 (Figure 1) during the first 72 hours of the hydration process. We considered three different compositions of concrete, called DYNA 0.5 and DYNA 0.33, with a water/cement (W/C) ratio of 50% and 33% respectively, and SCC 0.5, a self compacting concrete with a 50% W/C ratio. Details of the compositions are presented in Table 1.

Table 1: compositions of concrete (all units are in kg/m^3, except when mentioned otherwise)

	DYNA 0.5	DYNA 0.33	SCC 0.5
Sand (0/4)	670	625	696
Aggregates (4/14)	1280	1190	875
Water	150	150	175
CEM I 42,5 R	300	450	350
Plastifier Rheobuild 2000 PF (cc)	1500	4500	1200
Filler 2001 MS (cc)	-	-	276



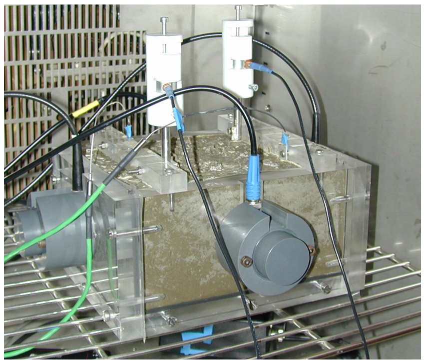


Figure 1: Cell used for monitoring the hardening process in young concrete, containing two pairs of compressional (P) and shear (S) transducers, two Acoustic Emission (AE) sensors and two temperature (T) sensors.

The cell contains a circular opening on each of the sides to fit four transducer holders for the active ultrasonic measurements. The opposite pairs of holders contain a transmitter and a receiver for either compressional (P) or for shear (S) wave investigations (Panametrics transducers, 25 mm active diameter, 0.5MHz and 0.25MHz central frequency respectively). A thin film closes the apertures and prohibits the freshly poured concrete from leaking through. Springs in the transducer holders ensure perfect contact between the transducers and the concrete. For the monitoring of the evolution of the inside temperature, two thermocouples are inserted from the (open) top side. Lastly, two very sensitive sensors (Vallen Systems, 2.05cm diameter, 0.375 MHz central frequency) attached on top of two protruding bars register the Acoustic Emission (AE) signals emerging from the microstructural activity in the concrete sample.

The measurements are performed in a temperature controlled room. After pouring the fresh concrete in the cubicle, the sample, with the sensors in place, is additionally isolated in a home-made measurement box (400×400×400 mm^3) to neutralize it as much as possible from the remaining external temperature and humidity fluctuations (mainly day-night cycles). The external (damped) temperature and humidity are monitored constantly and are used to correct the periodical fluctuations inside the concrete sample. The experimental set-up consists of two computer controlled systems. One system (Digital Wave) is registering the AE events, the second one performs the ultrasonic measurements (both linear and nonlinear) and the temperature reading. Both systems are independent from each other, except for the communication about the interruption of the registration at certain times. A LabVIEW script controls the acoustic measurements, adjusting the function generator (Agilent 33250A) and the oscilloscope (LeCroy 9310AM) settings through GPIB-IEEE488, and monitors the acoustic responses. Temperature (3 readings: inside temperatures $T_{in,1}$ and $T_{in,2}$, and outside temperature T_{out}) and relative humidity (RH) are logged on the same PC.

The AE events are registered continuously. At regular times after the start of the experiment (every 3 minutes in the beginning stages and every 7 minutes after the first day), the AE recording is interrupted by an active ultrasonic measurement and a reading of the condition parameters (temperature and humidity). For each of the wave polarizations (P and S), two types of ultrasonic measurements are performed: a pulsed wave and a continuous wave train transmission. In the first experiment, we apply a unipolar pulse of 1 μs to the respective transducer. From the arrival time of the received pulses in the pulsed P and S wave transmission experiments, we determine the speed of the waves for longitudinal and shear polarization. In the continuous wave experiments, we typically use a sequence of 100 cycle bursts at 100 kHz and evaluate the second harmonic amplitude dependence on the excitation amplitude at the receiver as a function of time/hardening. During the ultrasonic measurements there is no AE registration. The total off-time is less than 30 seconds. The hardening process is monitored by both systems during the first 72 hours.

2. Typical monitoring results

Figure 2 gives an overview of the typical measurement results for the DYNA 0.33 composition The temperature change inside the concrete sample, calculated as the difference between inside and outside temperature (to correct for day-night cycle), is shown in the top figure. A relatively silent start is followed by a crucial temperature rise, reflecting the internal accumulation of heat due to chemical reactions. The first chemical reaction starts really early in the process, about 2 hours after the preparation of the concrete, and the increase lasts for about 12 hours. It is during this period that most clustering between the different particles is established, first between the smallest and later between the largest particles [1-3]. After reaching the hydration peak, the temperature decreases gradually back to room temperature during the subsequent mechanical setting phase. The temperature profiles agree well with adiabatic hydration experiments.

The second subfigure in Figure 2 illustrates the cumulative counts of the Acoustic Emission events, after automatic filtering out bad readings related to instrument noise. The observations can again be linked to the various changes in the hydration process [5]. A silent phase is observed at first, followed by an accelerated increase of counts which starts just prior to the main temperature change, i.e. the period of intense hydration activity. At this point, it is believed that the largest particles in the concrete are fully connected. The silence during the connection period of small particles is not easy to explain, but one of the reasons could be that the recording of high frequency pulses is hindered by the high attenuation in the slurry, and do not attain the threshold settings of the AE device. Even after the temperature peak is reached the accumulation of AE counts goes on. This reflects the mechanical setting (shrinkage) of the concrete during the hardening process.

For what concerns the elasticity properties, ultrasonic through-transmission measurements using separated pairs of P and S wave transducers allow to determine the P and S pulse mode velocities (Figure 2c). Using these values, the Young's Modulus and Poisson Ratio is calculated (Figure 2d). Obviously, we witness here the complete transition between a fluid-like medium (E = low and $v \approx 0.5$), and a real solid block of concrete ($E \approx$ 40 MPa and $v \approx 0.2$) in the course of one day. During the first part of the curing (earlier than 6 hours after the preparation) the attenuation is so high that it is impossible to transmit any sound wave through the sample. Once the transmission of shear acoustic wave energy is possible, we observe a steep rise of the elastic modulus which lasts almost up to the maximum temperature increase, and corresponds to the phase of connecting larger and larger particles (a similar effect can be observed in the Poisson Ratio). The steep change is followed by a gradual evolution to an asymptotic value. Actually, the inflection point in the Young's modulus and Poisson Ratio is situated just before the temperature peak, and indicates the transition between the grain connection phase and the pore-filling phase within the sample, which is controlled by the diffusion of water and ions through the hydrate layers [1-4].

In the NEWS experiment, we measured and analyzed the changes in the second harmonic spectral content of a 100kHz continuous wave in transmission as function of the response amplitude. Because of the large attenuation in the beginning of the curing process, we performed the continuous wave transmission experiments in a slightly modified curing cell with a smaller transmission distance (50 m). We quantify the nonlinearity parameter β by calculating the proportionality coefficient from the quadratic dependence relation $A_2=\beta(A_1)^2$, with A_2 the level of the second harmonic, and A_1 the fundamental amplitude

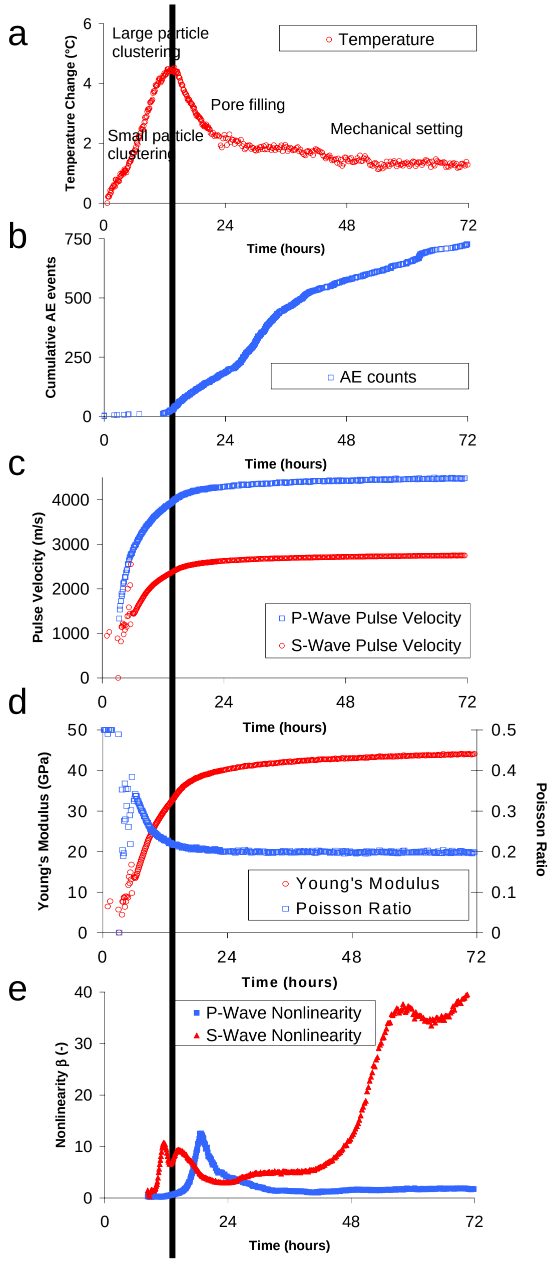


Figure 2: Typical monitoring results for the hydration process of DYNA 0.33 during the first 3 days: From top to bottom: evolution of temperature, cumulative AE events, P and S wave velocities, Young's Mode and Poisson Ratio, and P and S nonlinear elasticity.

[6-10,13]. The evolution of β, shown as function of the curing time in bottom subfigure of Figure 2 for both polarizations, tells us how the micromechanical nonlinearity in the stress-strain relation of the young concrete is changing during the hardening process. In general, the attenuation is too large in the "fluid" phase and the transmission intensity too small to generate measurable nonlinear effects. However, as the chemical activity in the concrete develops and the percolation threshold is reached (just before the peak in temperature), we observe a significant increase of the transversal (S-wave) nonlinearity. The increase reflects the micromechanical friction which is generated through the partial connection of the particles. As soon as the connections become better and better, the shear nonlinearity decreases, and so does the temperature. The development of the longitudinal (P-wave) nonlinearity is delayed with respect to the shear nonlinearity, and manifests itself primarily during the late chemical activity (the capillary pore filling stage) and in the early

mechanical creep (creation of shrinkage microcracks which can be activated by the pressure waves). In the subsequent phase we observe again an increased contribution of the shear nonlinearity. This time we attribute the raise to the increased mechanical shrinkage which is changing the stress state inside the concrete sample significantly and is creating microcracks with large enough openings to be susceptible to nonlinear shearing. Once the cracks are too wide open and under too high stresses, the nonlinearity cannot be activated anymore by the dynamic waves and we expect a subsequent drop at later times. Thus, in general, we may conjecture that the origin of the nonlinear components is connected to the micromechanical changes in the composition, both due to chemical reactions (mostly shear nonlinearity) and to progressive mechanical setting of the sample (longitudinal and shear).

Another way of interpreting the above data is to represent them as function of the degree of hydration, a significant parameter representing the fraction of hydrated products during the hardening process. Analytical models have been developed to calculate the degree of hydration based on the measured temperature evolution, the composition of the concrete sample, and its strength properties [14-15]. Figure 3 illustrates the hydration as function of time, and the representation of the measured results for DYNA 0.33 in terms of the hydration parameter. We found that the hydration process is most intense for a degree of hydration near 0.2, which corresponds exactly with the instance when the internal temperature increase is highest and the AE events start to appear. We also remark that the first appearance of shear nonlinearity can be observed close to the percolation threshold where the compression strength starts to develop, and thus provides information about the microstructural activity even sooner than for the AE readings.

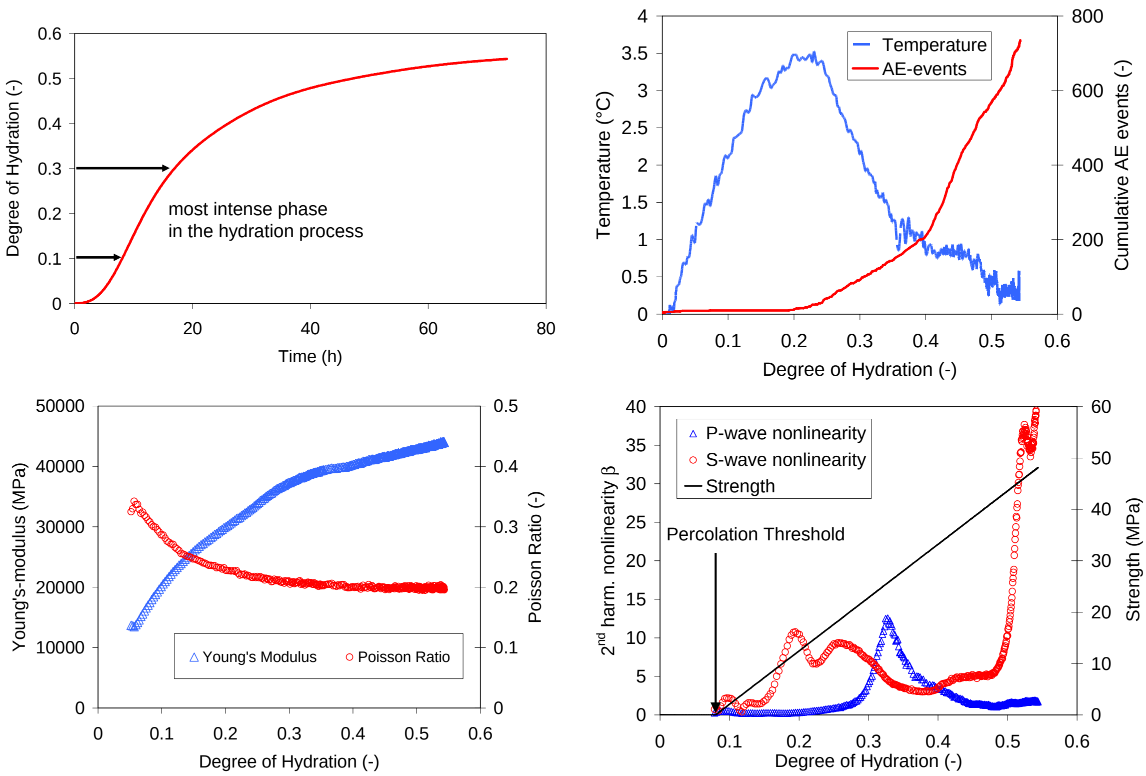


Figure 3: Evolution of the degree of hydration with time, and parametric representation of the variation in internal temperature, cumulative AE events, Young's Modulus and Poisson Ratio, and nonlinear elastic coefficients in DYNA 0.33 as function of the degree of hydration.

2. Influence of initial concrete composition

In Figure 4, we grouped the monitoring results for each of the three considered initial compositions of concrete (see Table 1). In terms of the temperature, the influence of the W/C ratio can be found in a reduction of the magnitude of the temperature peak (4°C for DYNA 0.33 and 2°C for DYNA 0.5) and a retardation of the peak values for larger W/C ratios. The peak width for SCC 0.5 appears to be more pronounced than for the DYNA 0.5 concrete even though they both have the same W/C ratio. This reflects the larger amount of hydration products in SCC. In addition, we see that the microstructural activity near the temperature peak for DYNA 0.33 changes at a more pronounced rate than for DYNA 0.5. The higher volume of water in the initial composition apparently slows down and smoothens out the activity during the mechanical setting process. For SCC 0.5, the AE reading starts earlier than for DYNA 0.5 since the chemical activity is more pronounced (cfr. temperature), but the acoustic emission during the mechanical setting is less intense.

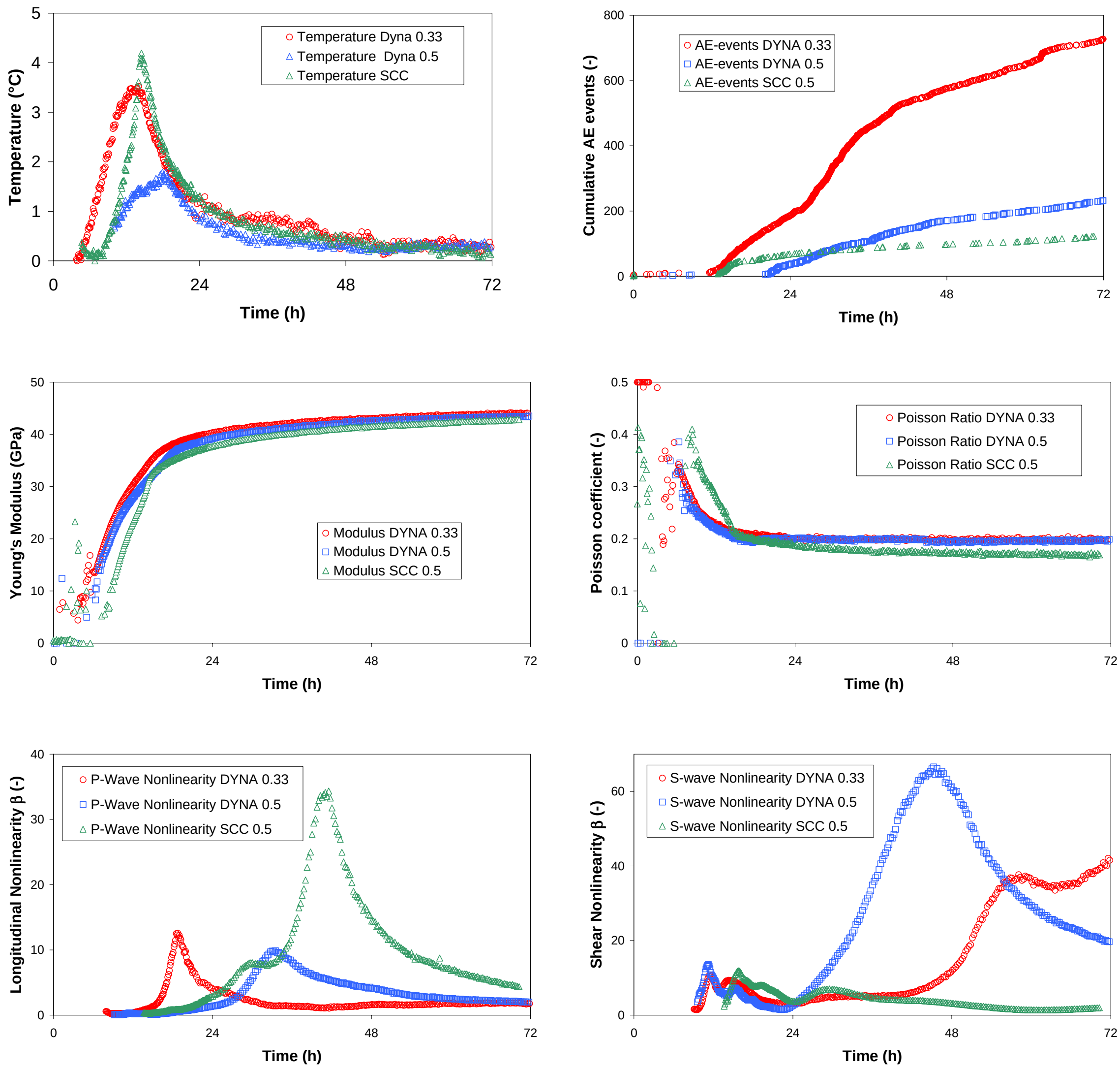


Figure 4: Comparison of the evolutions of the internal change in temperature, the cumulative AE events, the Young's Modulus and the Poisson Ratio, and the P and S wave nonlinearity in DYNA 0.5, DYNA 0.33 en SCC 0.5 during the first 3 days of the curing process.

The difference in the active ultrasonic wave monitoring results for different W/C ratios is not spectacular if we focus on the linear readings of P and S wave velocity and their deduced elasticity properties. A larger W/C ratio causes a slight retardation on the development of elasticity. Also, the steep increase of the temperature for SCC is translated in a steeper change of the linear elastic properties too. In terms of the nonlinear readings, we observe that the development of the nonlinearity deduced in the longitudinal wave propagation experiments is held back for DYNA 0.5 and SCC 0.5 with respect to the observations in DYNA 0.33. This can be attributed to the relative larger amount of water in DYNA 0.5 and SCC 0.5, and the relative lower amount of solid hydration products which postpones the setting and delays the measurable nonlinearity to later times. Also significant is the observation that the shear nonlinearity in SCC is completely suppressed during the mechanical setting, whilst the longitudinal nonlinearity shows up late but nevertheless in a significant manner. In view of this, we can conclude that, upon hardening, and more specifically in the mechanical setting, SCC is confronted with a rather uniform distribution of many and more importantly extremely tiny microcracks which generate more nonlinearity in compression than in shear. For the other compositions the cracks are most probably larger, and not uniformly present, which allows the shear nonlinearity to dominate.

Acknowledgments

The authors gratefully acknowledge the support of the Flemish Fund for Scientific Research. (G.0206.02, G.0257.02, and G.0554.06).

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