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Active and passive monitoring of the early hydration process in concrete using linear and nonlinear acoustics

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ABSTRACT

Microstructural changes occurring in freshly poured concrete during curing have been monitored on a laboratory scale using a combination of the Acoustic Emission (AE) Technique with linear and nonlinear ultrasonic/elastic wave spectroscopy. The AE technique is a passive ultrasonic signal recording technique capable of online monitoring the internal microstructural activity of young concrete during the hydration process. Ultrasonic wave spectroscopy is traditionally used to evaluate the material's longitudinal and shear wavespeed and attenuation properties (providing properties such as Young's Modulus of Elasticity, Poisson's Ratio and Quality factor) by means of an active excitation of a medium with pulsed sound waves. In addition to these traditional techniques, we have implemented a nonlinear version of ultrasonic wave spectroscopy which probes the nonlinear elastic properties of the microstructure (offering information about the micromechanical behaviour) through the analyses of the harmonic generation from a continuous wave transmission through the concrete sample. The evolution in the AE events, and in the linear and nonlinear ultrasonic behaviour of young concrete is analyzed as a function of the degree of hydration for various initial compositions during the first three days of the curing process. The results show a good correlation between the linear and nonlinear acoustic properties and the phase changes in the concrete due to chemical reactions and mechanical setting seen in the temperature profile.

1. Introduction

It is known that the durability of cement based products and concrete structures is highly influenced by the early stages of hydration. The creation of an interfacial transition zone between the aggregates and the cement paste, with a thickness of up to 50 µm, is considered to be the origin of primary defects in concrete leading to preferred paths for crack propagation and transport of aggressive agents threatening the durability of concrete. A precise knowledge of the micro-mechanical properties during the successive phases of the hydration process will provide information on the concrete resistance and allows assessment of its durability. Several non-destructive techniques have been developed and applied in that respect, most of them based on ultrasonic wave measurements. Keating et al. [1] observed a three stage evolution in the velocity of longitudinal waves in cement slurries after mixing. Many other researchers have observed similar patterns using ultrasonic pulse velocity measurements [2–14], and related these results, in combination with other physical parameters, to the hydration process. Boumiz [6,7] and Morin [11] used active ultrasonic echographic measurements providing the linear elastic material coefficients in combination with volumetric shrinkage measurements to describe the evolution of the capillary network of a High Performance Concrete during hardening. Feylessoufi et al. [12] determined a relation between the shear wave reflection coefficient and the percolation threshold of reactive powder concrete. Voigt et al. [13] investigated Portland cement mortar during hydration by means of the shear wave reflection method, and showed that the measurements are governed primarily by the degree of the inter-particle bonding of the cement particles as calculated from the specific contact area of a simulated microstructure. Passive energy recording using Acoustic Emission (AE) techniques has been used to evaluate the structural activity in concrete at early ages [14], showing periods of intense microstructural changes during the curing process. However, till now, the correlation between the linear elastic material properties, the number of AE-events and the micromechanical properties remains unclear, and a quantification of microstructural transformations (i.e. chemical and physical alterations and/or damage) induced by chemical reactions and setting during curing is not easily achieved.

The particular aspect of monitoring the influence of crack induced porosity on the micromechanics during hardening, may well be handled by nonlinear dynamic mechanical measurements. Nonlinear Elastic Wave Spectroscopy (or short NEWS) techniques represent a class of recently developed powerful tools which explore the dynamic

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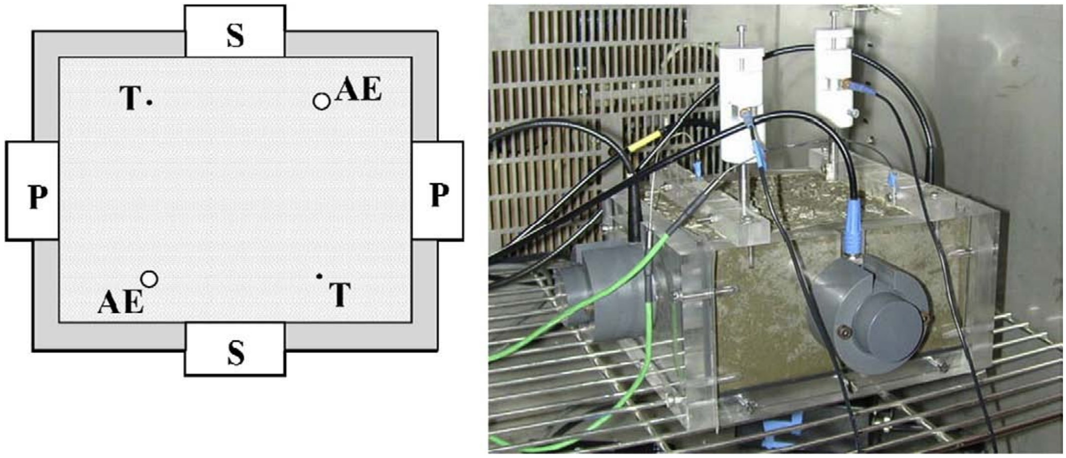

Fig. 1. Top view (left) and picture (right) of the curing cell used for monitoring the hardening process in young concrete. The monitoring devices include AE sensors (AE), thermocouples (T) and compressional (P) and shear (S) transducers.

nonlinear stress–strain features in the compliant bond system of a micro-inhomogeneous material and link them to micro-scale imperfections [15–21]. Micromechanical features such as hysteresis and elastic nonlinearity in the constitutive relation (at the micro-strain level) result in acoustic and 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 [19]. In a series of laboratory NDT applications on various materials [15,18,20,21], 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, linear ultrasonic wave propagation and NEWS harmonic monitoring. With this study, we complement and extend the work of Lacouture et al. [22] who started the use of NEWS for monitoring the first chemical reaction phase in the curing process. Here, we report NEWS results for the first three days of the hydration process. Using appropriate theoretical hydration models [23,24], we interpret these results within the framework of the degree of hydration concept. Eventually this work should lead to an improved prediction of the long-term behaviour of concrete and its performance dependence on the curing processes.

2. Setup for integrated passive and active monitoring experiments

2.1. Sample holder, instrumentation and concrete composition

The monitoring experiments were performed on a cubicle measuring $200 \times 150 \times 100$ mm^3 (Fig. 1) during the first 72 h of the hydration process. The cell contains a circular opening on each of the sides to fit four transducer holders for the active ultrasonic measurements: a transmitter and receiver for both compressional (P) and shear (S) waves. The compressional transducers (Panametrics, 25 mm active diameter, 0.5 MHz central frequency) are positioned on the cross side and have a separation distance of 200 mm. The shear transducers (Panametrics, 25 mm active diameter, 0.25 MHz central frequency), placed on the long side, have a mutual distance of 150 mm. A thin film closing the apertures 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 variation, two thermocouples are inserted from the (open) top side. Lastly, two very sensitive sensors (Vallen Systems, 20.5 mm 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.

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.

2.2. Integrated monitoring procedure

The monitoring measurements are performed in a temperature controlled room. After pouring the fresh concrete in the cubicle, the instrumented sample is placed in a climate chamber (Heraeus-Vötsch Climate Cabinet VLK 04/150) (Fig. 2) to isolate it from large outside temperature and relative humidity fluctuations. The climate chamber is closed. However, climate conditions are not actively controlled in order to allow and to monitor the variations of the concrete's inside temperature, which are characteristic for the concrete curing process. The (damped) temperature inside the climate chamber is also registered and is used to correct the thermal fluctuations in the concrete sample for day–night cycles.

Fig. 3 depicts the instrumentation scheme of the experimental setup, which 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.

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

Fig. 2. The sample is placed in a climate chamber, closed without active control, diminishing outside temperature and relative humidity fluctuations.

The AE events are registered continuously. At regular times after the start of the experiment (every 3 min in the beginning stages and every 7 min 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. The arrival time is determined using two techniques, providing similar results: cross-correlation and STA/LTA triggering (short-term-averaging/long-term-averaging [25]). The data set covering 72 h of monitoring is analyzed backwards in time (from hardened to freshly poured) to assure correct identification of the pulse arrivals and wave polarisation. In the continuous wave experiments, we typically use a sequence of 100 cycle bursts at 100 kHz and evaluate the generation of the second harmonic (200 kHz) during transmission of the wave through the evolving micro-inhomogeneous medium. If the medium is not micromechanically nonlinear, it will not produce harmonics. However, if the setting of the microstructure for instance allows nonlinear or hysteretic contacts of grains and interfaces, these features will induce the generation of harmonics which can be macroscopically observed in the recorded wave signal [26,27]. According to the standard theory of classical nonlinear wave propagation, the amplitude of the second harmonic is quadratic in the amplitude of the excitation. The nonlinear parameter used in this study is determined as the proportionality coefficient in the quadratic second harmonic amplitude dependence on the excitation amplitude at the receiving transducer, and its evolution is monitored as a function of time/hardening. During the ultrasonic measurements there is no AE registration. The total off-time is less than 30 s.

The hardening process is monitored by both systems during the first 72 h. Sequences, consisting of registering condition parameters and acoustic responses, are repeated every 3 min in the first 12 h of the experiment. Between 12 h and 24 h, sequences are repeated every 7 min. After 1 day, measurements are performed every 15 min. AE events are registered continuously, except at times when acoustic waves propagate through the medium.

3. Results and analysis

3.1. Typical monitoring results

Fig. 4 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 h after the preparation of the concrete, and the increase lasts for about 12 h. It is during this period that most clustering between the different particles is established, first between the smallest and later between the largest particles [4,6]. After reaching the 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 same subfigure of Fig. 4 also 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 [4,14]. 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.

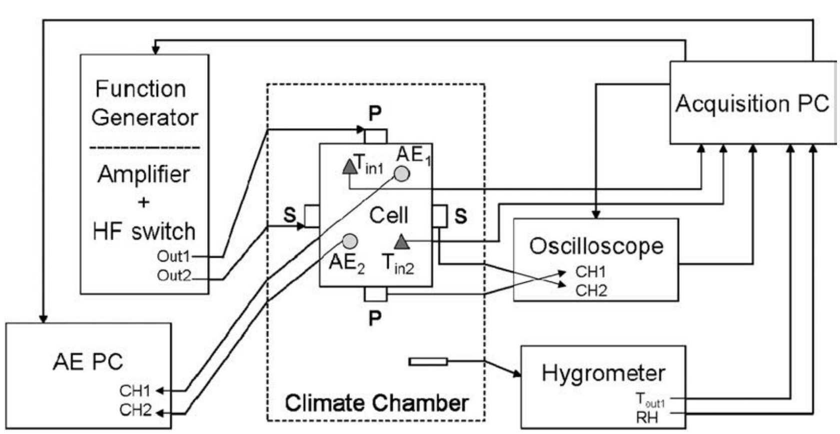

Fig. 3. Experimetal setup: the system registering AE events is largely independent from the system monitoring condition parameters and acoustic response.

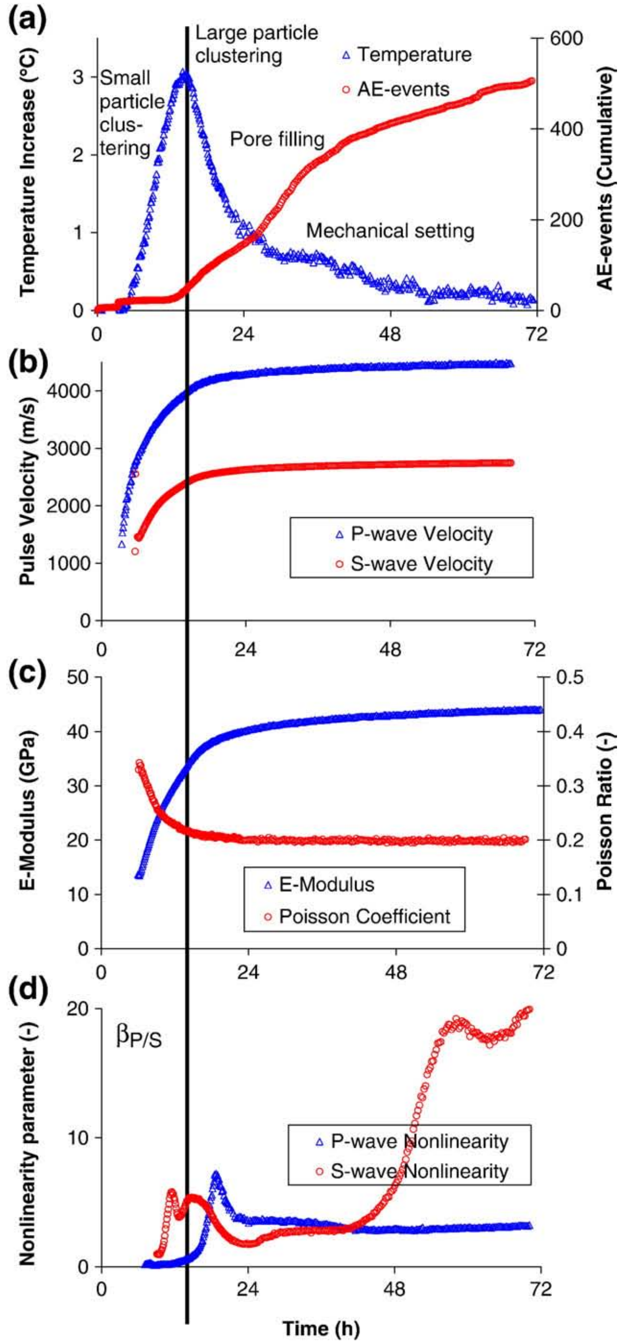

Fig. 4. Typical monitoring results for the hydration process of DYNA 0.33 during the first 3 days: From top to bottom: evolution of temperature (a), cumulative AE events (a), P and S wave velocities (b), Young's Modulus and Poisson Ratio (c), and P and S nonlinear elasticity (d).

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 using cross correlation or STA/LTA triggering (Fig. 4b). Using these values, the Young's Modulus (E) and Poisson Ratio (ν) is calculated (Fig. 4c). Obviously, we witness here the complete transition between a fluid-like medium (E = low and $\nu \approx 0.5$), and a real solid block of concrete (E ≈ 40 MPa and $\nu \approx 0.2$) in the course of one day. During the first part of the curing (earlier than 6 h 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. This stage 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 very near 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 [4,6].

In the NEWS experiment, we measured the second harmonic spectral content generated by a 100 kHz continuous wave in transmission as function of the response amplitude, and monitored the evolution of the nonlinear parameter during the hardening process. Changes in the micromechanical properties of the medium due to nonlinear contact forces, frictional contacts and sliding grain boundaries will be reflected in a macroscopically observable change in the nonlinear parameter [26,27]. 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 mm). As mentioned above, we quantify the nonlinearity parameter β by simply 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 [15–22]. The evolution of β, shown as function of the curing time in the bottom subfigure of Fig. 4 for both polarizations, provides an indication 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. We postulate that 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, as 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 rise 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 gather that the observed variations of the nonlinear components can be logically 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).

3.2. Analysis in terms of degree of hydration

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 [23,24]. Fig. 5 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

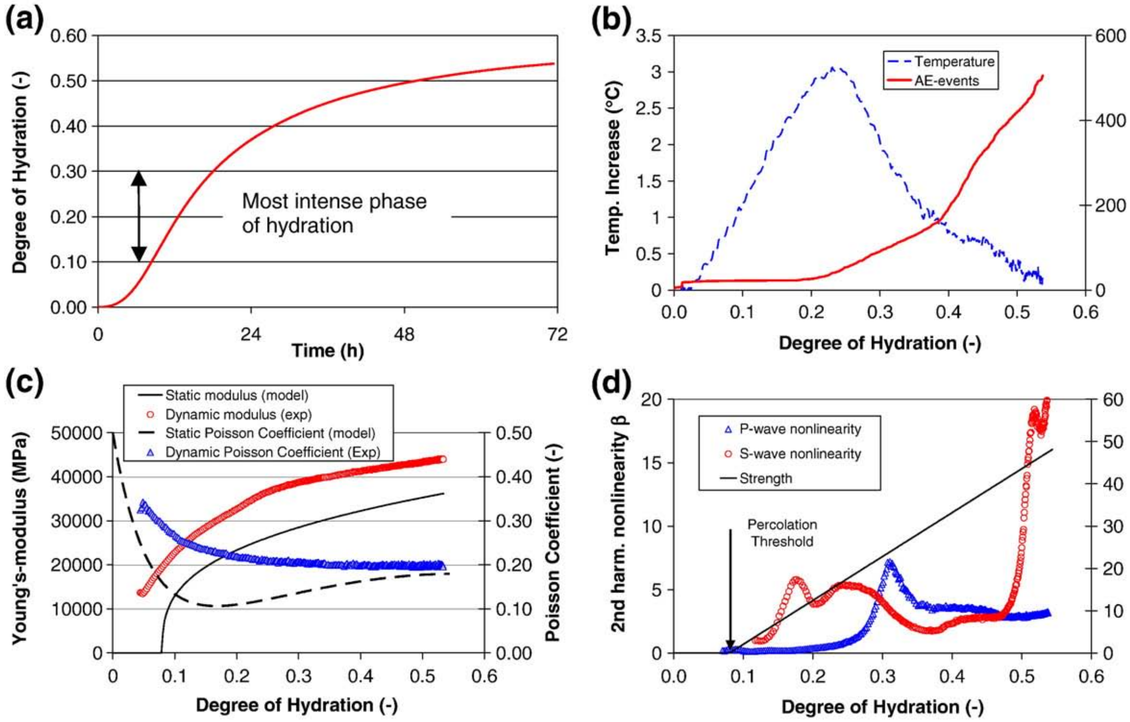

Fig. 5. Evolution of the degree of hydration with time (a), and parametric representation of the variation in internal temperature (b), cumulative AE events (b), Young's Modulus and Poisson Ratio (c), and nonlinear elastic coefficients (d) in DYNA 0.33 as function of the degree of hydration.

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. The agreement between the overall evolution of the (calculated) static young's modulus and the experimentally obtained dynamic Young's modulus is striking. It is known that dynamic modulus values are always higher than static ones, which explains the "offset". Apart from the minimum predicted by the hydration model, the evolution of the dynamic Poisson's Ratio is also comprehensible. We finally 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.

3.3. Influence of initial concrete composition

In Figs. 6–9, we grouped the monitoring and analysis results for the three considered initial compositions of concrete (see Table 1). Fig. 6 illustrates the degree of hydration as function of time for the various compositions. There is a slight deviation between the three compositions after reaching the temperature peak, i.e. in the pore filling phase.

Fig. 7 compares the temperature change and AE-events for the three compositions. 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 (3 °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 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.

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 (Fig. 8). 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 as well.

In terms of the measured nonlinearity parameter (Fig. 9), we observe that the development of the nonlinearity deduced in the longitudinal wave propagation experiments is delayed 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

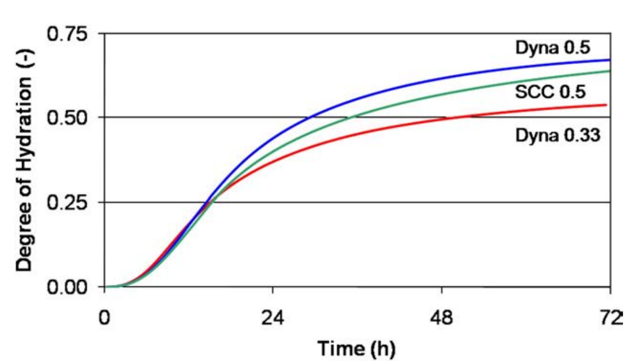

Fig. 6. Degree of hydration versus time for different concrete compositions.

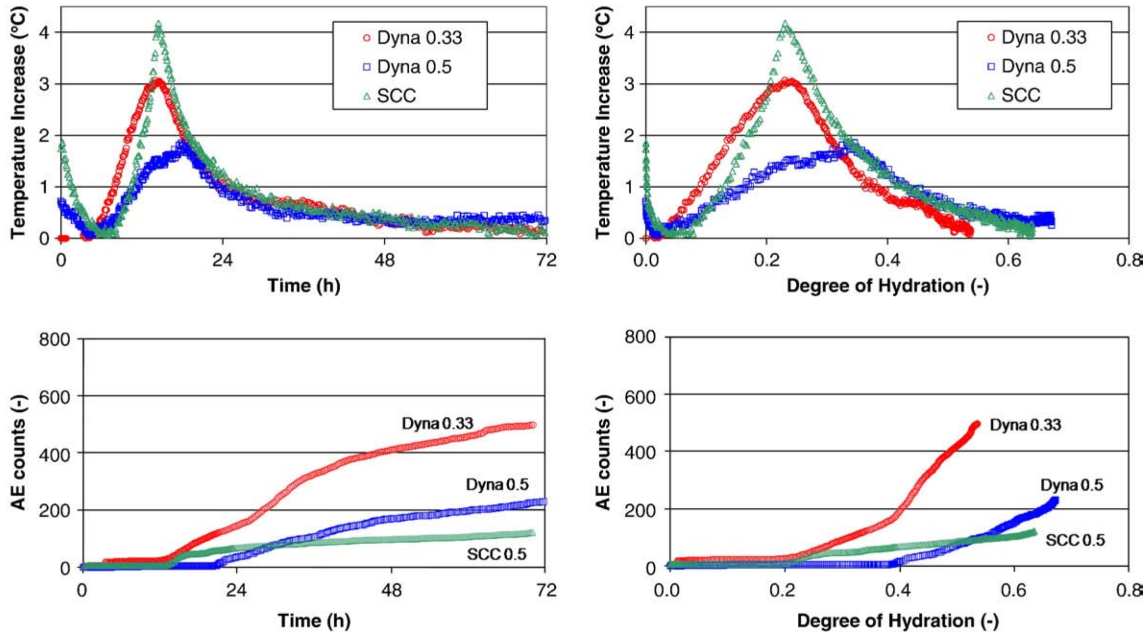

Fig. 7. Comparison of the evolutions of the internal temperature change (a and b) and the cumulative AE events (c and d) in DYNA 0.33, DYNA 0.5 en SCC 0.5 during the first 3 days of the curing process as function of time (left) and as function of degree of hydration (right).

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 and contact surfaces 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. It is also striking that certain features which are happening at different times apparently happen at the same degree of hydration. This can be observed for the P-wave nonlinearity of DYNA 0.5 and SCC 0.5, and for the S-wave nonlinearity in DYNA 0.33 and DYNA 0.5, which suggests that the nonlinearity parameters are indeed connected to the micromechanics of the microstructure of the young concrete.

4. Conclusion

In this paper, the results of an integrated study are reported to monitor the hardening process in concrete by combining temperature condition measurements, Acoustic Emission (AE) readings and linear and nonlinear ultrasonic/elastic wave spectroscopy. The purpose of this study was to link the measurements to the microstructural changes occurring in freshly poured concrete and during the

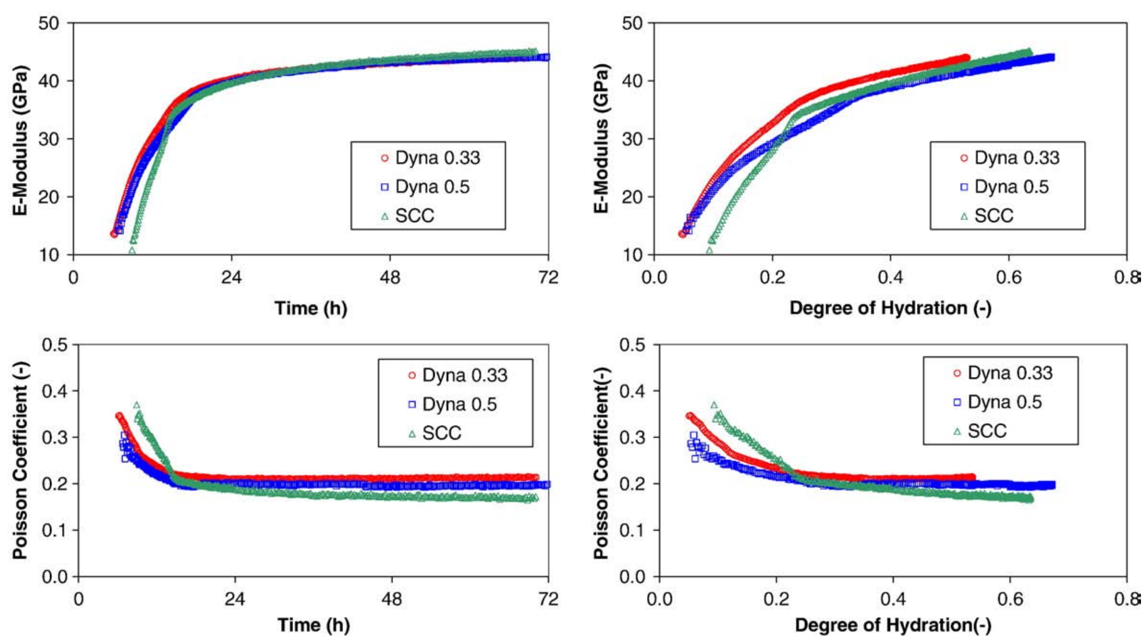

Fig. 8. Comparison of the evolutions of the Young's Modulus (a and b) and the Poisson Ratio (c and d) in DYNA 0.33, DYNA 0.5 en SCC 0.5 during the first 3 days of the curing process as function of time (left) and as function of degree of hydration (right).

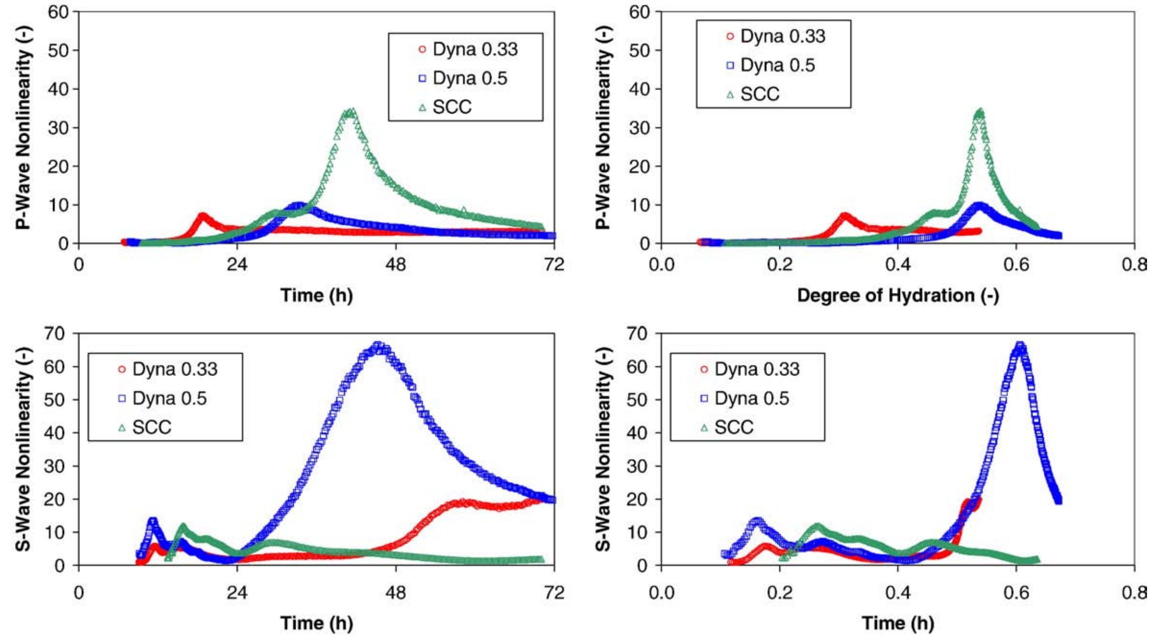

Fig. 9. Comparison of the evolutions of the P wave nonlinearity (a and b) and S wave nonlinearity (c and d) in DYNA 0.5, DYNA 0.33 en SCC 0.5 during the first 3 days of the curing process as function of time (left) and as function of degree of hydration (right).

subsequent curing. Beyond the traditional measurement data, specific attention was drawn on the interpretation of the nonlinear ultrasonic measurements. It was found that different features in the evolution of the nonlinear parameters can be logically connected to the micro-mechanical changes in the structure, both due to chemical reactions (mostly shear nonlinearity) and to progressive mechanical setting of the sample (longitudinal and shear), thereby providing additional information that is hidden in other monitoring results.

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References

[1] J. Keating, D.J. Hannant, A.P. Hibbert, Comparison of shear modulus and pulse velocity techniques to measure the buildup of structure in fresh cement pastes used in oil well cementing, Cem. Concr. Res. 19 (1989) 554–566.
[2] J. Keating, D.J. Hannant, A.P. Hibbert, Correlation between cube strength, ultrasonic pulse velocity and volume change for oil well cement slurries, Cem. Concr. Res. 19 (1989) 715–726.
[3] S. Popovics, R. Silva-Rodriguez, P.S. Popovics, V. Martucci, Behavior of ultrasonic pulses in fresh concrete, New Experimental Techniques for Evaluating Concrete Materials and Structural Performance, SP-143, ACI, Detroit, MI, USA, 1993, pp. 207–225.
[4] C.M. Sayers, A. Dahlin, Propagation of ultrasound through hydrating of cement pastes at early times, Adv. Cem. Based Mater. 1 (1993) 12–21.
[5] V. Garnier, G. Corneloup, J.M. Sprauel, J.C. Perfumo, Setting time study of roller compacted concrete by spectral-analysis of transmitted ultrasonic signals, NDT E Int. 28 (1995) 15–22.
[6] A. Boumiz, Etude comparée des évolutions mécaniques et chimiques des pâtes de ciment et mortiers a très jeune age. Développement des techniques acoustiques, Thèse de Doctorat de l'Université Paris 7, (1995) Paris.
[7] A. Boumiz, C. Vernet, F. Cohen Tenoudji, Mechanical properties of cement pastes and mortars at early ages, evolution with time and degree of hydration, Adv. Cem. Based Mater. 3 (1996) 94–106.
[8] H.W. Reinhardt, C.U. Grosse, A.T. Herb, Ultrasonic monitoring of setting and hardening of cement mortar – a new device, Mat. Struct. 33 (233) (2000) 581–583.
[9] M.I. Valic, Hydration of cementitious materials by pulse echo USWR – method, apparatus and application examples, Cem. Concr. Res. 30 (10) (2000) 1633–1640.
[10] A. Smith, T. Chotard, N. Gimert-Breart, D. Fargeot, Correlation between hydration mechanism and ultrasonic measurements in an aluminous cement: effect of setting time and temperature on the early hydration, J. Eur. Ceram. Soc. 22 (12) (2002) 1947–1958.
[11] V. Morin, F. Cohen Tenoudji, A. Feylessoufi, P. Richard, Evolution of the capillary network in a reactive powder concrete during hydration process, Cem. Concr. Res. 32 (2002) 1907–1914.
[12] A. Feylessoufi, F. Cohen Tenoudji, V. Morin, P. Richard, Early ages shrinkage mechanisms of ultra-high-performance cement-based materials, Cem. Concr. Res. 31 (2001) 1573–1579.
[13] T. Voigt, G. Ye, Z. Sun, S.P. Shah, K. van Breugel, Early age microstructure of Portland cement mortar investigated by ultrasonic shear waves and numerical simulation, Cem. Concr. Res. 35 (2005) 858–866.
[14] D. Bonnet, K.El. Kouhen, Etude de la microfissuration des Bétons par émission acoustique, rapport de maîtrise de physique, Lab, Lab. Dir. Scient. Bouygues, vol. 7, LED Université Paris, 1998.
[15] L. Adler, P.B. Nagy, Second order nonlinearities and their application in NDE, in: D.O. Thompson, D.E. Chimenti (Eds.), Review of Progress in Quantitative Nondestructive Evaluation, vol. 10B, Plenum, New York, 1991, pp. 1813–1820.
[16] O. Buck, W.L. Morris, J.N. Richardson, Acoustic harmonic generation at unbonded interfaces and fatigue cracks, Appl. Phys. Lett. 33 (5) (1978) 371–373.
[17] V.E. Nazarov, A.M. Sutin, Nonlinear elastic constants of solids with cracks, J. Acoust. Soc. Am. 102 (1997) 3349–3354.
[18] K. Van Den Abeele, P.A. Johnson, A.M. Sutin, Nonlinear Elastic Wave Spectroscopy (NEWS) techniques to discern material damage, Research in Nondestuctive Evaluation 12/1 (2000), 17-30 &31-42.
[19] R. Guyer, P. Johnson, Non linear mesoscopic elasticity: evidence for a new class of materials, Phys. Today 52 (1999) 30–35.
[20] K. Van Den Abeele, J. De Visscher, Damage assessment in reinforced concrete using spectral and temporal nonlinear vibration techniques, Cem. Concr. Res. 30/9 (2000) 1453–1464.
[21] K. Van Den Abeele, K. Van De Velde, J. Carmeliet, Inferring the degradation of pultruded composites from dynamic nonlinear resonance measurements, Polym. Compos. 22 (4) (2001) 555–567.
[22] J.C. Lacouture, P.A. Johnson, F. Cohen Tenoudji, Study of critical behavior in concrete during curing by application of dynamic linear and nonlinear means, J. Acoust. Soc. Am. 113 (2003) 1325–1332.
[23] G. De Schutter, L. Taerwe, General hydration model for portland cement and blast furnace slag cement, Cem. Concr. Res. 25 (3) (1995) 593–604.
[24] G. De Schutter, L. Taerwe, Degree of hydration based description of mechanical properties of early age concrete, Mat. Struct. 29 (1996) 335–344.
[25] See http://psn.quake.net/sdrdocs/stalta.htm.
[26] V.A. Aleshin, K. Van Den Abeele, Micro-potential model for stress-strain hysteresis of micro-cracked materials, J. Mech. Phys. Solids 53 (4) (2005) 795–824.
[27] V.A. Aleshin, K. Van Den Abeele, Microcontact based theory for acoustics in microdamaged materials, J. Mech. Phys. Solids 55 (2) (2007) 366–390.