On-line monitoring of setting and hardening of concrete

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Summary

The paper deals with the ultrasonic transmission method to determine setting and hardening of cementitious materials. A testing device is described. An important feature of the method is the automatic signal evaluation. To this end, the continuous wavelet transform has been applied. Measurements on mortar with shotcrete accelerator demontrate the applicability of the method. Results are discussed. Further research needs are outlined.

1. Motive

Until recently, laboratory tests on cement pastes with the Vicat needle were normally used to define standardized initial and final setting times [1]. However, these paste tests have been critizised for providing results distinct from those observed in the field [2, 3]. Moreover, the selection of these two points in the continuous process of cement hydration is rather arbitrary. More recently, the constant depth penetrometer has been used to evaluate the compressive strength of cast in place concrete for strength up to 1 MPa. During the last decade other non-destructive techniques have attracted attention for the characterisation of the behaviour of concrete at early age. Among these, ultrasonic pulse velocity measurements permit to continuously follow microstructure development in concrete and mortar at early age [4, 5, 6, 7, 8]. The ultrasonic pulse velocity measurements are related to the development of the modulus of elasticity and the Poisson ratio. A correlation with more traditional methods such as penetration has been established [9]. The ultrasonic test methods have the great advantage that they are continuously monitoring the development of mechanical properties of mortar and concrete.

2. Ultrasound transmission

2.1 Principle and device

The ultrasound device used for the current investigations was the FreshCon developed at the University of Stuttgart and described in more detail in earlier publications (among others [10, 5, 11, 12]). The container consists of two polymethacrylate (PMMA) walls which are tied together with four screws with spacers (Fig. 1). The mould is a U-shaped rubber foam element with high damping properties, suppressing waves from travelling through the mould and thus around the mortar. The volume of the mould is approximately 30 cm^3. At one side of the mould a pulse is generated using a broadband frequency generator (National Instruments), an amplifier (PI) and an ultrasound transmitter (Panametrics).

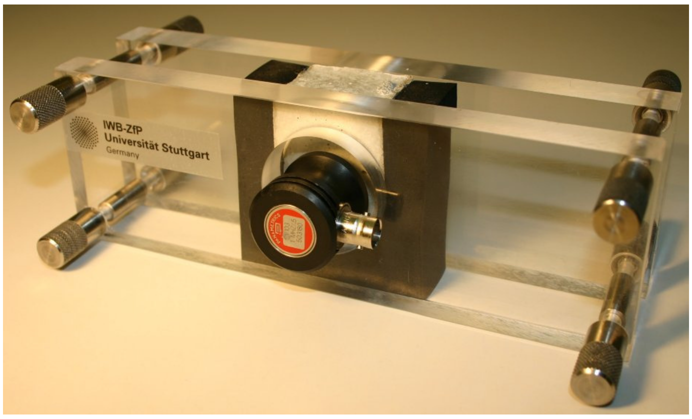


Fig. 1. View of the FreshCon mortar container

After travelling through the mortar sample in the mould, the signal is recorded at the other side by an ultrasound receiver (Panametrics), with a sampling rate of 20 MHz. Preliminary tests showed that the change in ultrasound velocity and energy could be adequately monitored using a recording interval of 0.5 min during the first half hour and a recording interval of 2 to 5 min later on. Before the experiment, the FreshCon device was calibrated both with an empty container and the two PMMA plates coupled, and with a reference sample with known travel time of the P-wave in between. The calibration parameters obtained were a time delay of 3.18 µs and a reference energy of 968.21 x 10^{-6}. The time delay is the time the ultrasound wave needs to travel through the sensors and the container walls. It has to be subtracted from the measured time to calculate the ultrasound velocity in the mortar sample. Furthermore, the ultrasound energy, determined by numerical integration of the squared amplitude values following the trigger time (which correlates to the onset), is divided by the reference energy and presented as a dimensionless value. The FreshCon software shows the received ultrasound signals and their frequency spectrum (using an FFT-algorithm) online during the experiment. Also the change in ultrasound velocity and energy and the frequency content vs. concrete age are represented. An offline version of the software allows re-evaluating the data after the test, using different algorithms for picking the onset times of the signals. Reinhardt and Grosse [5] determined that, between repetitions, measured velocities vary only by approximately 1%.

2.2 Signal evaluation

Furthermore, a new automatic onset picking algorithm was tested offline [14, 22]. The onset detection by hand is a very time consuming procedure, however it is also important for calculating correct velocities. Manual onset detection is performed by inspecting the transmitted time signal on the computer screen and selecting the first measurement point deviating from the noise. A reliable auto-picker should determine values close to the ones gained by handpicks and the shape of e.g. the velocity vs. concrete age curves should be maintained.

Therefore, an adapted automatic picker based on the Akaike Information Criterian (AIC) is presented. It produces reliable results for acoustic emissions and for ultrasound signals with a relative high success rate [14]. The problem concerning acoustic emissions and ultrasound signals in concrete is that signal and noise are often in the same frequency range. Furthermore, due to failure processes in the tested specimen, the signal to noise ratio of acoustic emissions is generally not constant during an experiment. Zang et al. [13] successfully applied an automatic frequency based onset determination algorithm similar to the STA/LTA picker to acoustic emissions from rock samples. However, acoustic emissions from rock samples are mostly to be found in a higher frequency range than acoustic emissions from concrete. Fig. 2 shows two examples of signals of concrete of one test with a different signal to noise ratio. The use of anti-causal, zero phase filters or the careful use of the wavelet transform can help to improve the signal to noise ratio. Nevertheless, a reliable automatic picker which can handle data of varying quality is needed.

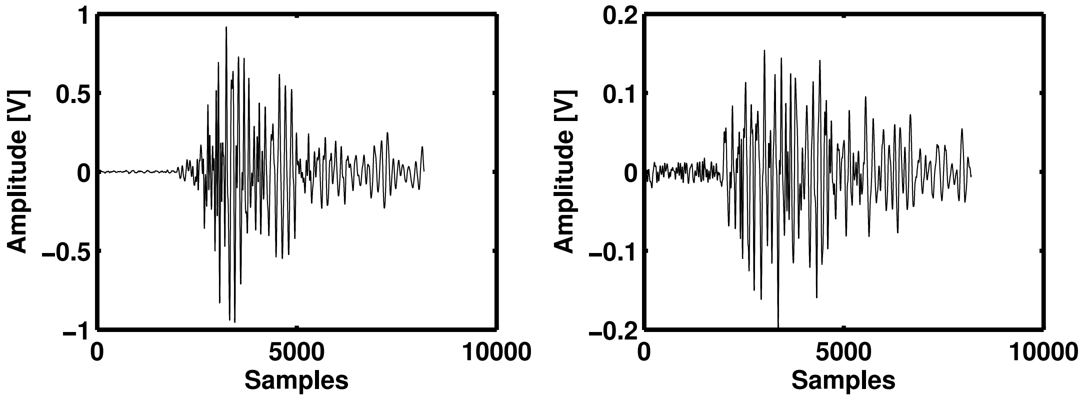


Fig. 2. Bandpass filtered emissions from one test with a different signal to noise ratio. The bandpass filter was an anti-causal zero phase butterworth filter (3 kHz, 110 kHz).

An autoregressive AIC-picker gives picks (picks means determined onset times) of higher quality if the AIC is only applied to a part of the signal which contains the onset, of course [15]. Therefore, the onset is prearranged by using the complex wavelet transform or the Hilbert transform. Both transforms lead to a certain envelope of the signal (Fig. 3). The Hilbert transform $\overline{R}(t)$ of a real time dependent function *R(t)* is defined as [16]:

$$\overline{R}(t) = \frac{1}{\pi}\int_{-\infty}^{\infty}\frac{R(u)}{t-u}du = \mathrm{H}\left\{R(t)\right\} \tag{1}$$

where *t* denotes the time and the singularity at $u = t$ is handled by taking the Cauchy principle value of the integral. The Hilbert transform is represented by a convolution integral, i.e. the Hilbert transform is a causal transfer function which behaves like a filter. Transforming a time series by the Hilbert transform, a phase shift of $\pi/2$ is generated. Thus, the envelope time function *E(t)* can be calculated according to Buttkus [16]:

$$E(t) = \sqrt{R(t)^2 + \overline{R}(t)^2} \tag{2}$$

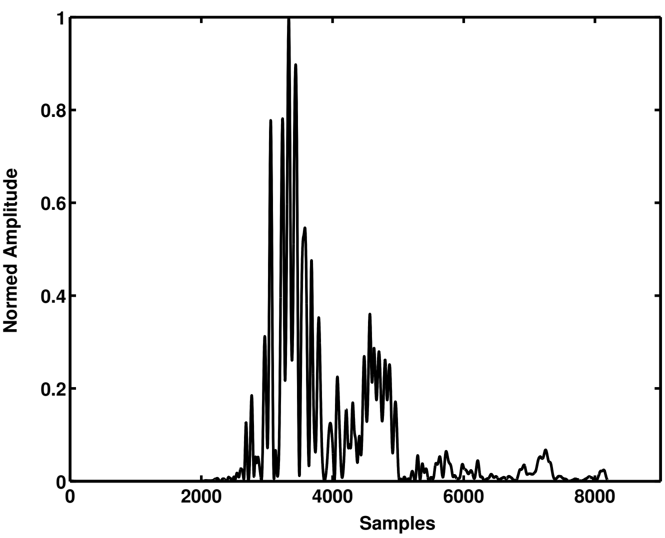


Fig. 3. Envelope of the signal calculated by the Hilbert transform

The complex continuous wavelet transform W of a discrete sequence $R(t)$ is defined as the convolution of $R(t)$ with a scaled and translated version of the wavelet function $\psi_{\lambda,\nu}$ [17]:

$$W(\lambda,\nu)=\int_{-\infty}^{\infty}\psi_{\lambda,\nu}(t)R(t)dt,\ \text{where}\ \psi_{\lambda,\nu}(t)=\frac{1}{\sqrt{\lambda}}\left(\frac{t-\nu}{\lambda}\right) \tag{3}$$

Continuous wavelet transform means continuously shifting a continuously scalable function $\psi_{\lambda,\nu}$ over the signal and calculating the correlation between the two. Thus, λ denotes the scale (scale is proportional to frequency) and ν the translation. The discrete sequence $R(t)$ is decomposed into a set of basis functions with the new dimensions λ and ν.

Since the complex continuous wavelet transform is a complex valued orthonormal transform represented by a convolution integral, the modulus of one scale of the complex continuous wavelet transform represents the envelope of an signal at one certain frequency.

$$|W(\lambda,\nu)|=\sqrt{x^2+y^2}\ \text{ where }\ W(\lambda,\nu)=x+iy \tag{4}$$

The advantage of the envelope calculated by the wavelet transform is that even for noisy signals the prearrangement of the onset by a threshold works steady. The envelope is calculated only for one scale while most of the noise of the signals is found in different scales.

However, if two or more signals of different amplitude and frequency superpose each other, i.e. if acoustic emissions occur in a very fast succession that more than one signal is recorded within the normal blocklength, the envelope calculated by the Hilbert transform should be used. Due to the automatic scaling, the wavelet transform can take the wrong signal for such a case.

The envelope is then used for prearranging the onset by a simple threshold. Each envelope is squared and normed, so that a constant threshold value can be applied to all signals. A window of several hundred samples e.g. 400 before and 150 after this point is then cut out of the signal. Within this part of the signal the onset is determined exactly using the AIC.

The exact onset is determined by calculating the AIC function direct from the signal according to Maeda [18]:

$$AIC(t_w)=t_w\cdot\log(\text{var}(R_w(t_w,1)))+(T_w-t_w-1)\cdot\log(\text{var}(R_w(1+t_w,T_w))) \tag{5}$$

The index w e.g. from R_w denotes that not the whole time series is taken but only the chosen window containing the onset (described above). T_w is the last sample of the curtate time series, t_w ranges through all samples of R_w and var denotes the variance function. The term $R_w(t_w,1)$ means that the variance function is only calculated from the current value of t_w while $R_w(1+t_w,T_w)$ means that all samples ranging from $1+t_w$ to T_w are taken. The sample variance var or σ_{N-1}^2 is defined as [19]:

$$\sigma_{N-1}^2=\frac{1}{N-1}\sum_{i=1}^{N}(R_i-\overline{R})^2 \tag{6}$$

N denotes the length of the signal, R_i is sample i of the time series R and $\overline{R}$ is the mean value of the whole time series R. The global minimum of the AIC function defines the onset point of the signal (Fig. 4).

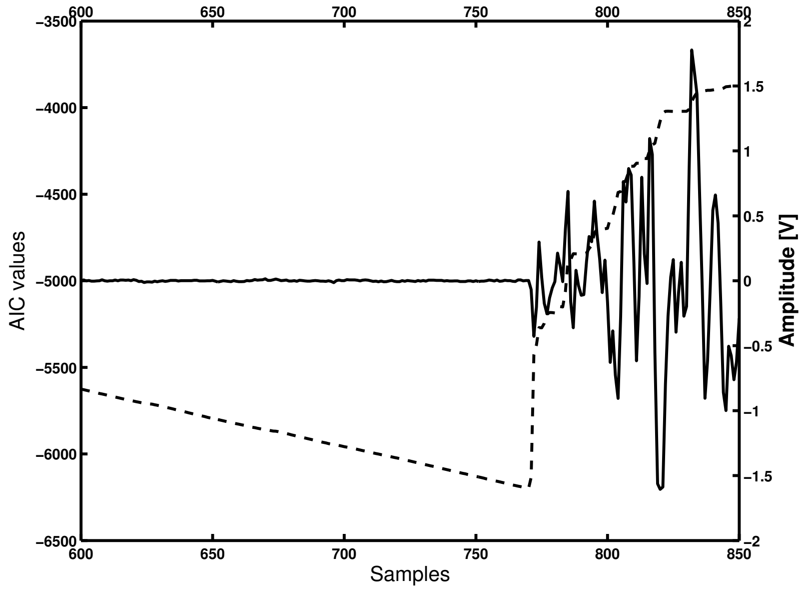


Fig. 4. The AIC is used for onset determination only for the selected part of the signal containing the onset which is displayed by the solid line. The minimum of the AIC function which is represented by the dashed line denotes the onset time of the signal

3. Measurements on mortar with shotcrete accelerator

To allow accurate measurements, starting rapidly after the mix preparation, the experiments were performed on mortar. Also the more traditional methods for monitoring concrete setting are mainly performed on cement paste or mortar. Due to practical limitations, the mortar cannot be pneumatically applied in the FreshCon container and a traditional compaction procedure was used instead.

The reference mortar consisted of 1350 g standard sand according to EN 196-3 [1] , 450 g cement, and 225 g water and was prepared according to EN 480-1 [20]. The cement types tested were a Portland cement CEM I 42.5 R and a Portland-limestone cement CEM II/A-LL 42.5 R including 6-20% limestone according to EN 197-1 [21]. The Portland-limestone cement is sometimes suggested as an alternative to Portland cement for shotcrete applications in Germany. The tested accelerators included an alkaline aluminate based solution (AlA) and an alkali-free solution based on aluminium sulphate (AlS). The accelerator dosage amounted to 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml accelerator per kg cement [22].

Cement and water were mixed for 30 s at low speed in a mixer in accordance with EN 196-3 [1]. Over the next 30 s at low speed, the dry sand was added, followed by 30 s mixing at high speed. After a rest period of 90 s, mixing was continued for a further 60 s at high speed. Then the accelerator was added and the mix procedure was concluded with about 5 s mixing at low speed and 5 s at high speed. The FreshCon container and three moulds for mortar prisms with dimensions 40 x 40 x 160 mm were filled and compacted for about 15 to 60 s on a vibrating table. The vibration time was limited in order not to hamper the binding process. The FreshCon container was sealed with plastic tape to allow cement hydration to proceed normally, and to avoid shrinkage of the mortar resulting in decoupling of mortar and container walls. The ultrasound measurements were started within 2 min after addition of the accelerator to the mix. The experiment was conducted at a room temperature of 20°C. The mortar prisms were stored in the sealed moulds at 20°C and demoulded after 24 h. Afterwards they were stored under water at 20°C until they were tested in bending and compression at an age of 28 days.

The results for ultrasound velocity and energy are shown in Figs. 5 and 6 respectively. For clarity separate graphs are shown for the two accelerator types. The different mortar mixes are coded as follows:

- I (CEM I 42.5 R) or II (CEM II/A-LL 42.5 R)
- AlA (alkaline aluminate based solution) or AlS (alkali-free solution based on aluminium sulphate)
- number representing the accelerator dosage relative to the maximum allowable dosage of 50 ml accelerator per kg cement (0, 0.5, 0.75 or 1)

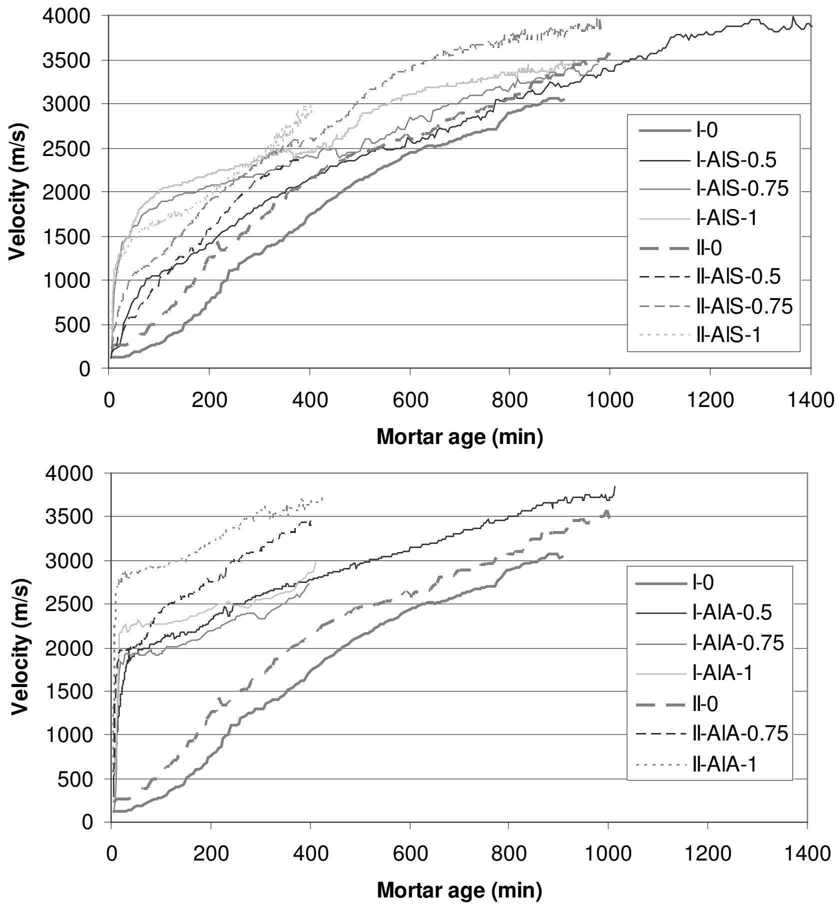
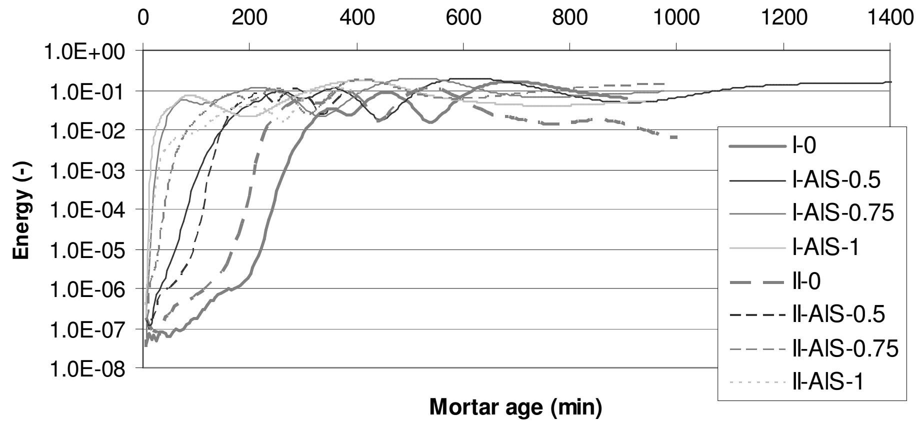


Fig. 5. Ultrasound velocity (hand picked onset time) vs. age for mortars containing the alkali-free accelerator AlS (top) or the alkali aluminate based accelerator AlA (bottom). Mortars were prepared using the cement types I 42.5 R (I) or CEM II/A-LL 42.5 R (II) and an accelerator dosage of 0, 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml per kg cement.



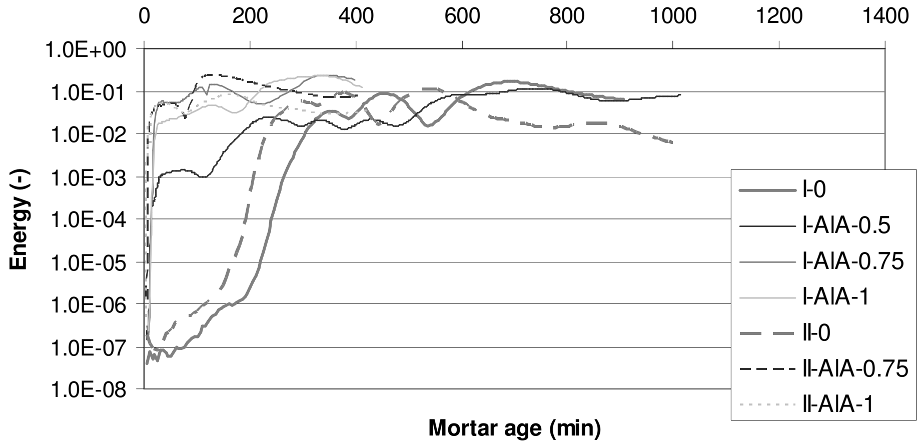


Fig. 6. Ultrasound energy vs. age for mortars containing the alkali-free accelerator AlS (top) or the alkali aluminate based accelerator AlA (bottom). Mortars were prepared using the cement types I 42.5 R (I) or CEM II/A-LL 42.5 R (II) and an accelerator dosage of 0, 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml per kg cement.

In all tested mixes the ultrasound velocity evolves from 100 to 700 m/s at a mortar age of 6 min (this is 6 min after adding the water to the mix, thus 2 min after accelerator addition) to about 4000 m/s at later ages. It is clear that the ultrasound measurements are sensitive to the effect of cement type, accelerator type and dosage on the binding and hardening behaviour of the mortar. A stepwise increase of the accelerator dosage resulted in increasing values for the pulse velocity at early ages. While non-accelerated mortar showed a dormant period of about 30 min before the pulse velocity started to increase sharply, no such threshold could be noticed in the accelerated mortar.

The alkaline accelerator AlA had an even more pronounced influence on the microstructure development than the alkali-free accelerator AlS, especially at ages below 90 min. Mortar accelerated with AlA was characterised by a very steep increase in pulse velocity during the first 15-30 min after which the velocity curve levelled off. For mortar with alkali-free accelerator AlS the velocity curve evolved more smoothly.

The effect of the alkali-free accelerator is at very early age more pronounced on mortar containing CEM I in comparison with CEM II. However, at an age between 150 and 400 min, the curves for CEM I and CEM II mortars have an intersection. This indicates that from this point onwards the mortar with CEM II is in a further stage of microstructure development. The alkaline accelerator seems to have a larger influence on mortar containing CEM II, compared to mortar with CEM I (when the same accelerator dosage is used).

The energy curves all showed the same pattern: an increase from 10^{-6}-10^{-7} to 10^{-2}-10^{-1} after which 3 local maxima could be noticed. These local maxima are probably caused by the characteristics of the sensors used, and to not really contain relevant information. The rate of energy increase depended on the accelerator type and dosage, and based on the energy curves more or less the same classification of mixes could be made as based on the velocity.

Some examples of the change in frequency content of the transmitted ultrasound signal with age are shown in Fig. 7. Other mortar samples show a similar picture with peak frequencies at early age around 20 kHz, changing to peak frequencies of 50 kHz later on. The frequency contents of subsequent transmitted ultrasound signals are represented through colour codes on vertical lines in the graphs. Fig. 7 therefore visualises which frequencies are transmitted best at a certain time in the setting and hardening process. The age at which this shift in frequency content occurs depends again on the variables tested (accelerator type and dosage, and cement type). The use of the alkali aluminate based accelerator causes the frequency shift to happen at very early age (7 to 12 min). While for the alkali-free accelerator this shift occurs between 8 and 110 min depending mainly on the accelerator content. The non-accelerated mortars show a frequency shift at the age of around 200 min (Fig. 7).

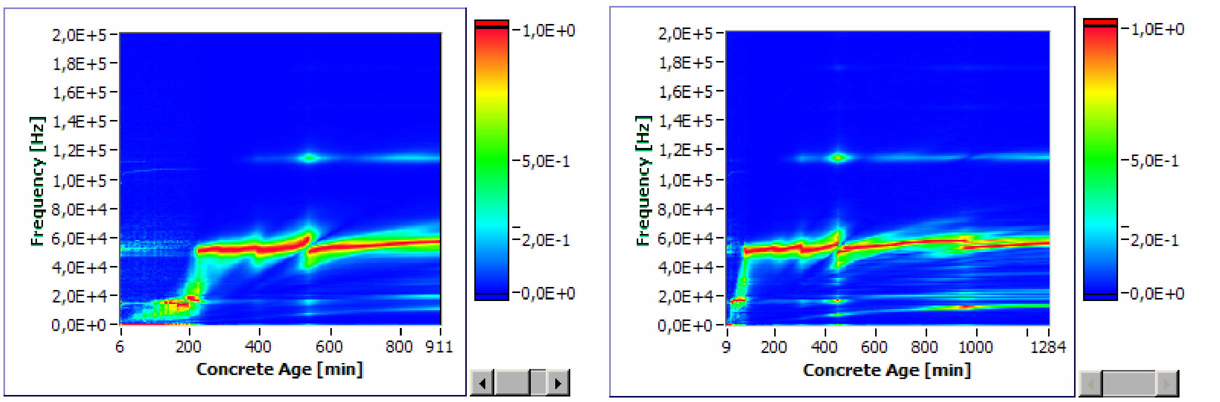


Fig. 7. Frequency content vs. age of the transmitted ultrasound signal for mortars I-0 (top) and I-*AlS*-0.5 (bottom)

4. Discussion

In unaccelerated samples with ordinary Portland cement, setting is normally completed within 6 to 7 h. Accelerators can shorten the setting time either by affecting the C_3A-hydration or by influencing the rate of C_3S-hydration. Initial CSH crystallization and the formation of CH (2-3 µm) generally contribute to the setting [23-25]. Also those ettringite crystals which are arranged radially on the clinker surfaces partially contribute in linking them and to a small extent to the setting of the sample. Paglia et al. [24] found that alkali-free accelerating admixtures (based on $Al_2(SO_4)_3.14H_2O$ with or without alkali-free calcium sulfoaluminate) promote the crystallization of ettringite prisms on the clinker surfaces at very early stage. The formation of ettringite prisms within the first 30 min is sufficient to set the samples and within 4 h of hydration, these crystals grow and almost fill the capillary pores between the clinker grains. In their research the use of an alkaline accelerator ($KAl(OH)_4$ aqueous solution) resulted in shorter setting times compared to the alkali-free accelerated samples. This is mainly due to precipitation of CH plates and amorphous KCAS$\bar{S}$H hydrates, rather than the formation of ettringite rods.

In our study the alkaline accelerator AlA resulted also in a faster velocity increase, and therefore a faster microstructure development than the alkali-free accelerator AlS, especially at very early ages (< 90 min). Paglia et al. [24] mention that the slightly longer setting times of the alkali-free admixture, compared to the alkali-rich admixture, appear to be favourable for the shotcreting efficiency. Due to the higher plasticity of the cementitious mass a better adhesion onto the tunnel wall is achieved, whereas the very fast setting attained by the alkali-rich admixture promotes a fast hardening of the cementitious mass, which in contact with the tunnel wall is easily rebounded.

According to prEN 934-5 [26] a requirement for sprayed concrete set accelerating admixtures is that the final setting time determined on reference mortar should be less than or equal to 60 min. Practical experience showed that the final setting time could be defined by an ultrasound velocity of 1500 m/s [5]. Our data confirm this statement, since for the reference mix with ordinary Portland cement (I-0) an ultrasound velocity of 1500 m/s was reached after 355 min which corresponds well with the setting period of 6 to 7 h, mentioned by [23]. Van der Winden [4] used a number of practical criteria to determine the limits of workability and found that the end of the workability was defined by the area where the ultrasound propagation speed increased from 1000 to 1500 m/s. The ultrasound velocity at 60 min is for the different experimental mixes presented in Fig. 8. If a velocity of 1500 m/s is taken to indicate the final setting time, the maximum dosage of alkali-free accelerator in combination with CEM II would just fulfil the requirements, while lower dosages would not be sufficient. In combination with CEM I, the dosage of alkali-free accelerator could be somewhat below the maximum dosage. For the alkaline accelerator even a dosage of half the maximum amount would suffice.

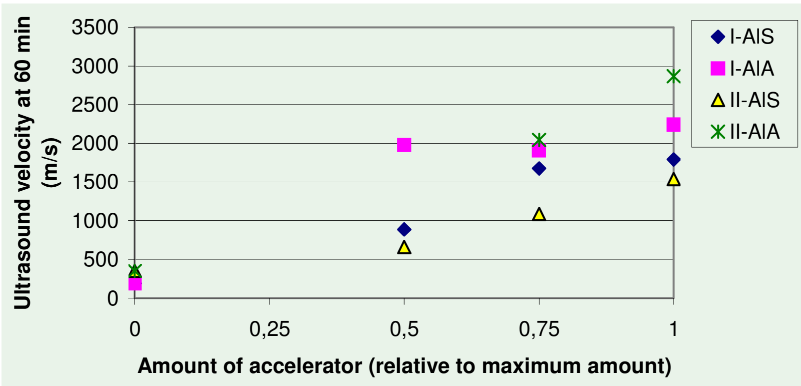


Fig. 8. Ultrasound velocity at 60 min for the different experimental mixes

Ye [7] found that the point where the pulse velocity started to increase sharply, could be indicated as a threshold of solid percolation. The cement hydrates then form a complete path of connected particles for the ultrasonic pulse wave. We found this dormant period to be around 30 min long in non-accelerated mortar, and non-existing (or shorter than the 2 min interval between accelerator addition and start of measurements) in accelerated mortar. The following quick increase of pulse velocity is caused by the quick change in connectivity of the solid phase. When all particles are connected, the slow increase in pulse velocity follows the evolution of the total solid fraction of the paste.

The energy change of the transmitted ultrasound wave is a parameter that has been less discussed in literature, mostly because it was difficult before to reproduce the energy of the ultrasound emittor. Close examination of the velocity and energy curves will learn that the first local maximum in the energy curve corresponds reasonably well to the age at which a velocity of 1500 m/s is reached. To be more exact, the first energy maximum is reached on average 20 min after the moment when the velocity equals 1500 m/s (Fig. 9). Therefore we could hypothesise that the energy is related to the setting phenomenon and that the maximum is reached when the end of workability is approached.

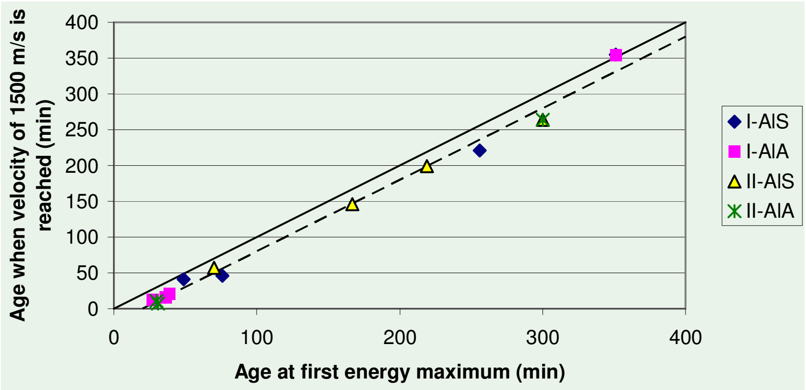


Fig. 9. Mortar age at which the ultrasound velocity of 1500 m/s is reached vs. age at first energy maximum. Straight lines represent y = x and y = x-20 (min)

Also the age at which the frequency shifts from about 20 to 50 kHz should have a physical meaning related to the setting phenomenon and pointing at a sudden increase in material stiffness. This point of frequency shift corresponded more or less to the moment when the rate of velocity increase was reduced. Comparing with the work of [7] this could correspond to the point where the cement hydrates form a fully connected solid frame. From this point onwards the "deceleration phase" starts and any further evolution of pulse velocity follows the evolution of the total solid volume fraction.

5. Outlook

The paper describes the current state of the test method and the device which has been developed. Further research is needed into the evaluation of the frequency content of the signals. Furtheron, the properties of fresh concrete with respect to workability should be investigated, i.e. the relation between the ultrasonic signals and rheological properties should be established.

Acknowledgement

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