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Ultrasound monitoring of the influence of different accelerating admixtures and cement types for shotcrete on setting and hardening behaviour

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Abstract

The possible use of ultrasound measurements for monitoring setting and hardening of mortar containing different accelerating admixtures for shotcrete was investigated. The sensitivity to accelerator type (alkaline aluminate or alkali-free) and dosage, and accelerator–cement compatibility were evaluated. Furthermore, a new automatic onset picking algorithm for ultrasound signals was tested. A stepwise increase of the accelerator dosage resulted in increasing values for the ultrasound pulse velocity at early ages. In the accelerated mortar no dormant period could be noticed before the pulse velocity started to increase sharply, indicating a quick change in solid phase connectivity. The alkaline accelerator had a larger effect than the alkali-free accelerator, especially at ages below 90 min. The effect of the alkali-free accelerator was at very early age more pronounced on mortar containing CEM I in comparison with CEM II, while the alkaline accelerator had a larger influence on mortar containing CEM II. The increase of ultrasound energy could be related to the setting phenomenon and the maximum energy was reached when the end of workability was approached. Only the alkaline accelerator caused a significant reduction in compressive strength and this for all the dosages tested.

Keywords: Shotcrete; Acceleration; Cement; Admixture; Workability

1. Introduction

Since its first use, the number of practical applications for shotcrete has continued to increase. Shotcrete is employed in particular for repair works, for immediate temporary support of tunnel walls following excavations in unstable ground, for stabilisation of bridges and for concreting in difficult locations, such as abutments, undersides of beams and interiors of chimneys [1,2]. It is also particularly useful for structures with a special architectural shape, such as arches and curved forms. Important basic requirements are adequate adhesion to the substrate, satisfactory shooting stiffness enabling build-up of thicker sections and preventing dangerous fallout of fresh material from walls and overheads, and high early strength. In this context, setting accelerators become especially important. Chemically, accelerating admixtures for concrete can be divided into four major groups [3]: alkaline silicates, alkaline earth metal carbonates/hydroxides, sodium and potassium aluminates, and alkali-free accelerators often based on aluminium sulfate or calcium sulfoaluminate. A side effect of the traditional alkali-rich accelerators is a significant reduction of the ultimate strength (typical values for strength reduction at 28 days range between 20% and 50%) [3]. Also important are the health hazards associated with handling alkalis. A new generation of alkali-free accelerators was therefore introduced for the improvement of the mechanical parameters, working conditions, safety, lower environmental

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impact, and easier maintenance of existing tunnel facilities [4]. The effect of accelerators on the early strength depends basically on their chemical base, dosage and temperature. Since they act chemically, their performance is closely related to the cement employed, its chemical composition, fineness, and the presence of other possible shotcrete additives [5]. A change in any of these parameters may jeopardize this interaction; therefore it is necessary to determine in each case the compatibility between cement and accelerator.

One of the main difficulties in the study of accelerators performance is to define a method to monitor the shotcrete initial stiffness and strength evolution. Until recently, laboratory tests on cement paste with Gillmore needles [6] or a Vicat needle were normally used to define standardized initial and final setting times [7]. However, these paste tests have been criticised for providing results distinct from those observed in the field [5,8]. 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 strengths up to 1 MPa. The constant energy penetrometer enables assessment of compressive strengths up to 10 MPa [9]. 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 monitor microstructure development in concrete and mortar at early age [10–14]. The ultrasonic pulse velocity measurements are related to the development of the modulus of elasticity and the Poisson ratio [10]. [14] showed that the reflection of ultrasound wave energy was sensitive to the presence of admixtures. [15] stated that the ultrasonic pulse velocity measurements could be used to monitor the microstructure development during setting and hardening of mortar and concrete. A correlation with more traditional methods such as pin penetration or heat evolution has been established [16].

2. Aim of the research

The aim of the current research was to investigate the possible use of ultrasound measurements for monitoring binding and hardening of shotcrete. The sensitivity to changes in accelerator type and dosage, and the effect of accelerators in combination with different cement types was evaluated.

3. Materials and methods

3.1. Wave transmission measuring 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 [17,11–13]). Separate devices are available for concrete and mortar, and the latter was used here. 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 45 cm^3. At one side of the mould a pulse is generated using a broadband frequency generator (Hameg), an amplifier (Develogic) and an ultrasound transmitter (Vallen VS 30). After travelling through the mortar sample in the mould, the signal is recorded at the other side by an ultrasound receiver (Vallen VS 30), 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 968.21×10^{-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. [11] determined that, between repetitions, measured velocities vary only by approximately 1%.

Furthermore, a new automatic onset picking algorithm was tested offline. The onset detection by hand is a very

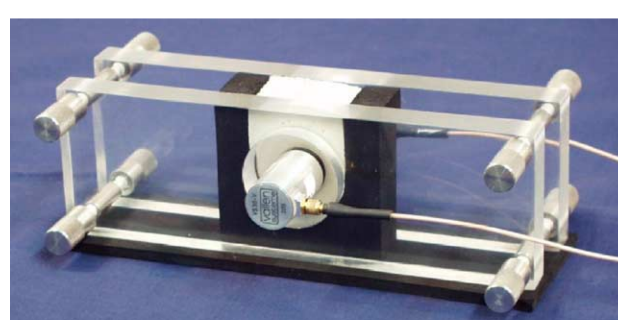

Fig. 1. View of the freshCon mortar container.

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. A detailed description of the used auto-picker can be found in [18]. Here, only a short description of the principle of the auto-picker will be given.

The onset time is determined iteratively by two steps. In the first step a certain part of the signal containing its onset is prearranged. Therefore, the normed envelope of the signal is used. The exact onset time is determined using the Akaike Information Criterion (AIC) after [19] within the prearranged window. The AIC means that a signal can be divided into locally stationary segments each modelled as an autoregressive process, i.e. the intervals before and after the onset time are assumed to be two different stationary time series. The global minimum of the AIC function defines the onset point of the signal.

3.2. Preparation of mortar specimens

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 196-1 [20], 450 g cement, and 225 g water and was prepared according to EN 480-1 [21]. 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 [22]. 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 sulfate (AlS). The accelerator dosage amounted to 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml accelerator per kg cement.

Cement and water were mixed for 30 s at low speed in a mixer in accordance with EN 196-1 [20]. 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 $40 \times 40 \times 160$ 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.

4. Results

The results for ultrasound velocity and energy are shown in Figs. 2 and 3 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 sulfate);
- 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).

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

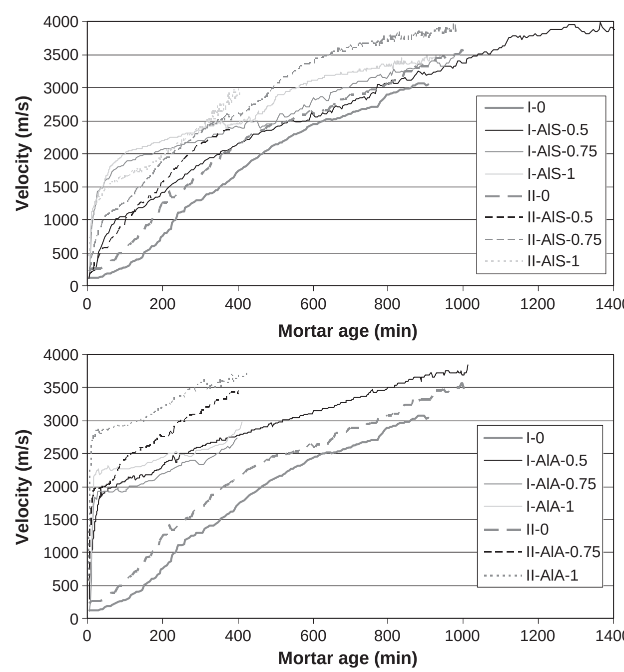

Fig. 2. Ultrasound velocity (handpicked onset time) vs. age for mortar containing the alkali-free accelerator AlS (top) or the alkali aluminate based accelerator AlA (bottom). Mortars were prepared using the cement types I 42.5 R (I) or CEM II/A-LL 42.5 R (II) and an accelerator dosage of 0, 0.5, 0.75 or 1 time the maximum allowable dosage of 50 ml per kg cement.

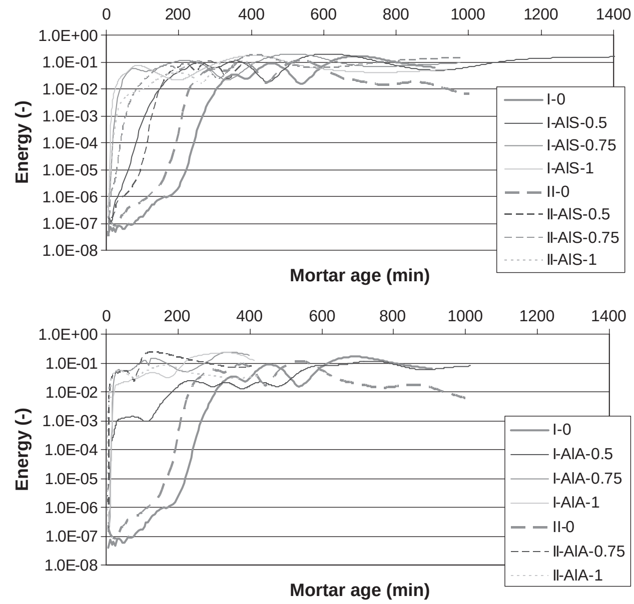

Fig. 3. Ultrasound energy vs. age for mortars containing the alkali-free accelerator AlS (top) or the alkali aluminate based accelerator AlA (bottom). Mortars were prepared using the cement types I 42.5 R (I) or CEM II/A-LL 42.5 R (II) and an accelerator dosage of 0, 0.5, 0.75 or 1 time the allowable dosage of 50 ml per kg cement.

Table 1
Mean deviation and maximum deviation of the auto-picker from handpicked velocity values investigated for four samples of the shotcrete II-AlS series

Sample	II-0	II-AlS-0.5	II-AlS-0.75	II-AlS-1.0
Mean deviation	3%	4%	2%	4%
Max. deviation	166 m/s	140 m/s	243 m/s	237 m/s

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 velocity vs. concrete age curves determined by the auto-picker based on the AIC criterion process were situated close to the handpicked curves (Fig. 4); i.e. the deviation of the auto-picker from the manual picks is small (Table 1) and the shape of each handpicked curve is similar to the auto-picker curves (Fig. 4). Considering that handpicks also

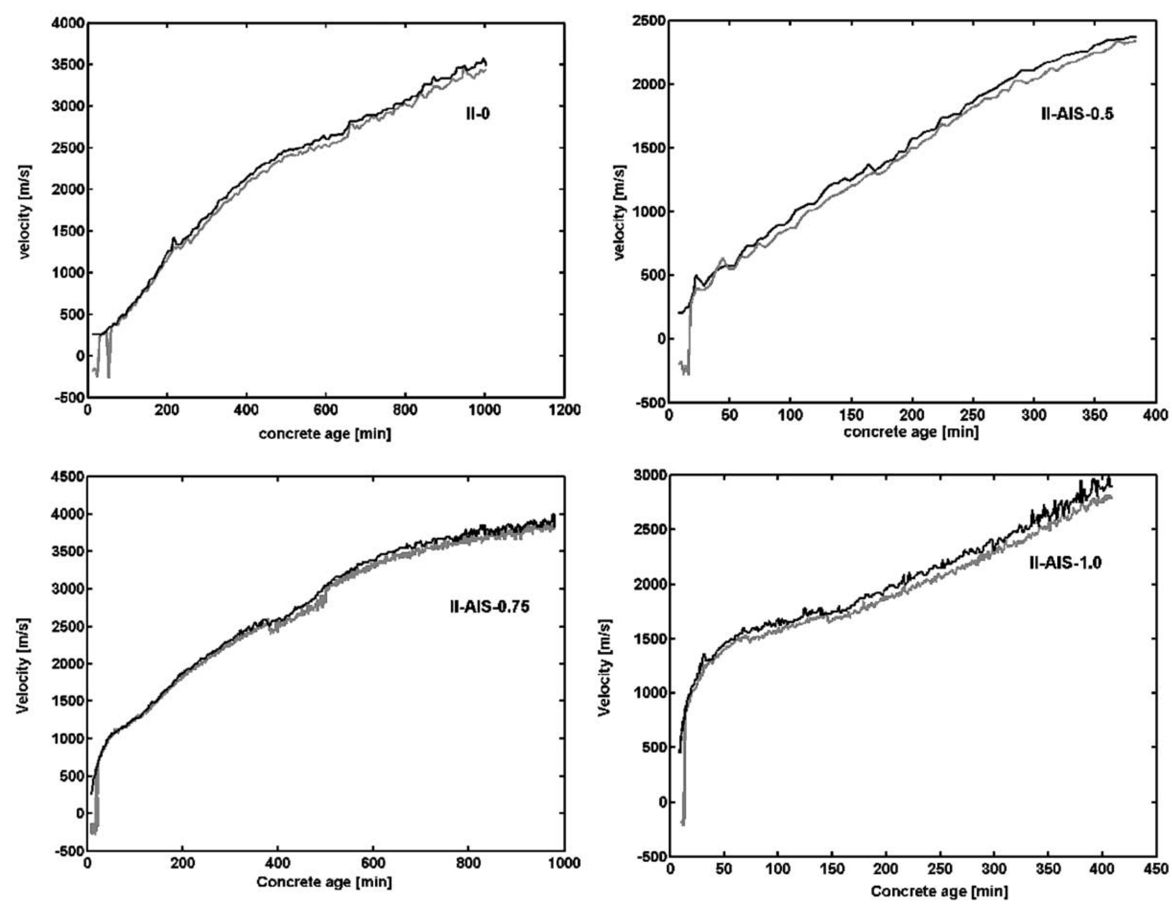

Fig. 4. Ultrasound velocity vs. concrete age for the II-AlS series, obtained by manual picking (black) or auto-picking) (grey) of the onset times.

contain a certain error due to mispicks, the mean deviation of the auto-picker of max. 4% from handpicks can be treated as low. However, due to the low signal to noise ratio of the first 10 to 15 measurements, the auto-picker makes a few false picks for these signals (see samples II-0 and II-AlS-0.5). These events have not been taken into consideration for the statistics of Table 1. Filter algorithms could help to avoid such false picks, however they have not been tested yet. The auto-picker has shown its reliable applicability to ultrasound signals for the monitoring of binding and hardening of concrete, here shotcrete. The results of the used auto-picker show that the time consuming handpicks can be set aside. The few mispicks within the first 10 to 15 signals are very striking, so that even a correction of these values by hand is possible. However, correct picks of all signals due to upstream filter algorithms are highly preferable. Different approaches are therefore under construction.

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 do 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. 5. 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. 5 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

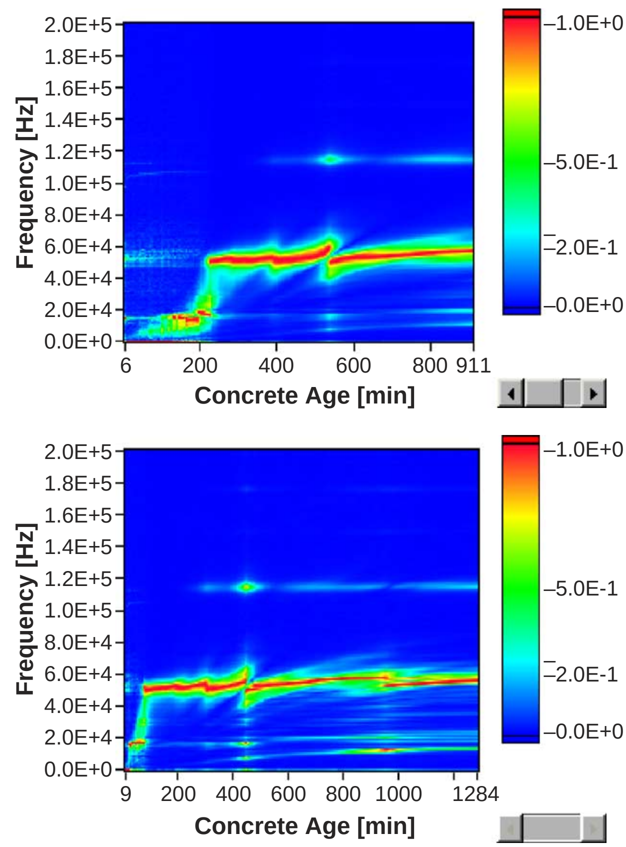

Fig. 5. Frequency content vs. age of the transmitted ultrasound signal for mortars I-0 (top) and I-AlS-0.5. (boottom).

(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. 5).

Fig. 6 shows the effect of accelerator type and dosage on the compressive strength, tensile strength in bending and density of the mortar prisms at 28 days of age. Only the alkaline accelerator AlA caused a significant reduction in the compressive strength in comparison with the reference without accelerator, and this for all the dosages tested (t-test with level of significance $p = 0.01$). The strength reduction was as high as 38% in combination with CEM I and 55% in combination with CEM II, when the maximum amount of accelerator was applied. This could be partly explained by an increase in the void volume, since the mortar density decreased significantly for these mixes e.g. with about 3% for the II-AlA-1 mix. For the tensile strength in bending there was a significant reduction with the addition of both accelerator types in dosages of 75% and 100% of the maximum amount. However, the reduction was much more drastic for the alkaline accelerator (about 30% with a 75% dosage and 35% with the 100% dosage) than for the alkali-free

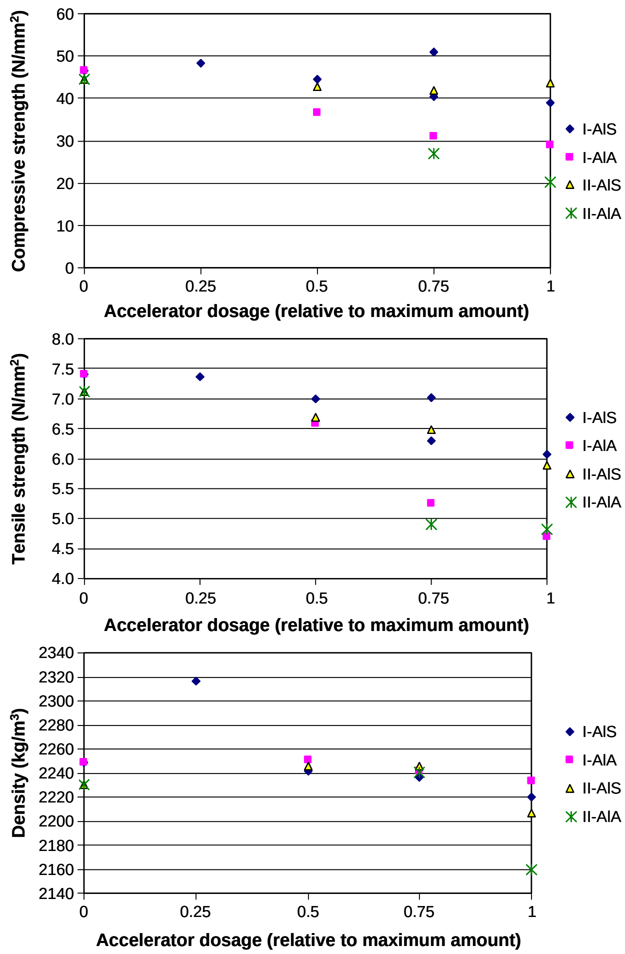

Fig. 6. Effect of accelerator type and dosage on the compressive strength, tensile strength in bending and density of the mortar prisms at 28 days (average standard deviation = 2.0 N/mm^2 for the compressive strength, 0.18 N/mm^2 for the tensile strength and 4.8 kg/m^3 for density).

accelerator (about 6% with a 75% dosage and 18% with the 100% dosage).

5. 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 [1,4,23]. 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. [4] found that alkali-free accelerating admixtures (based on $Al_2(SO_4)_3{\cdot}14H_2O$ 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 micro-structure development than the alkali-free accelerator AlS, especially at very early ages (<90 min). [4] 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 [24] 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 [11]. 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 [1]. [10] 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. 7. 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.

[15] found that the point where the pulse velocity started to increase sharply, could be indicated as a threshold of solid

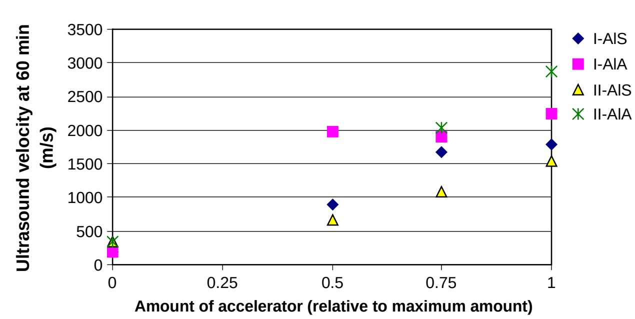

Fig. 7. Ultrasound velocity at 60 min for the different experimental mixes.

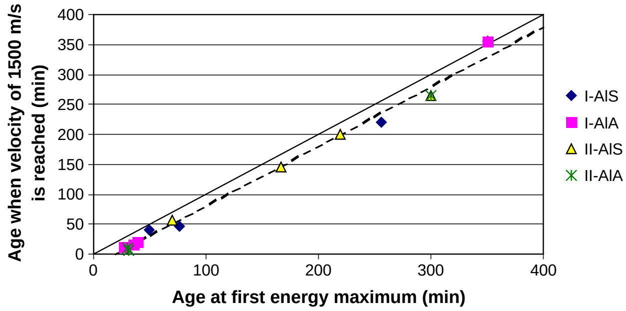

Fig. 8. Mortar age at which the ultrasound velocity of 1500 m/s is reached vs. age at first energy maximum. Straight lines represent $y = x$ and $y = x - 20$ (min).

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 mm/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. 8). 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.

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 [15] 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.

The results confirm that the strength decrease at later age is less severe for the alkali-free than for the alkaline accelerator. The decrease in compressive strength of the mix with the alkali-free accelerator was even not statistically significant when compared with a non-accelerated reference mortar. This was also found by [25] and [3]. [2,3,5] mention a decrease in 28-day compressive strength of 20–25% when an alkali aluminate based accelerating admixture is applied. In our study the strength reduction was even larger and amounted to 38% for mortar with CEM I. This could be due to an increase in void volume in our specimens, which was not experienced by [3]. A decrease in density of shotcrete with aluminate based admixtures was also described by [26], who suggested that the flash-setting of shotcrete indicated a certain loss in self-compaction ability. We experienced indeed for some of the mixes with high accelerator dosage, that by the time that the moulds of the mortar prisms were filled, further compaction became very difficult, since the setting process had already started. However, this does not necessarily imply that the same density decrease will occur in the field, since in practical applications the shotcrete is applied very fast on the substrate.

6. Conclusion

The ultrasound measurements were clearly sensitive to the effect of cement type, accelerator type and dosage on the setting behaviour of 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 (related to the quick change in connectivity of the solid phase), no such threshold could be noticed in the accelerated mortar. The alkaline accelerator had a larger accelerating effect on the microstructure development than the alkali-free accelerator, especially at ages below 90 min. The effect of the alkali-free accelerator was at very early age more pronounced on mortar containing CEM I in comparison with CEM II, while the alkaline accelerator had a larger influence on mortar containing CEM II. The mean deviation of maximum 4% between velocities calculated using the auto-picker and those based on manual onset time detection could be treated as low. The increase of ultrasound energy could also be related to the setting phenomenon and the maximum energy was reached when the end of workability was approached. Only the alkaline accelerator caused a significant reduction in compressive strength in comparison with the reference without accelerator, and this for all the dosages tested.

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