Mar. 2011, Volume 5, No. 3 (Serial No. 40), pp. 278-283
Journal of Civil Engineering and Architecture, ISSN 1934-7359, USA

Application of Ultrasonic Measurements for Determination of Setting and Hardening in Cement Paste

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Abstract: Concrete setting and hardening processes are the most critical phases during construction works, influencing properties of a concrete structure. The initial set is important as it provides an estimate when concrete has reached a point that it can no longer be vibrated without damaging concrete. The point at which final set occurs is important since it provides an estimate when the development of concrete strength and stiffness starts. In this study, experimental work is performed under laboratory conditions to indentify the setting time of cement paste using ultrasonic waves. Correlation between the ultrasonic wave parameters (speed, amplitude and energy) passing through the fresh cement paste and setting time determined using the Vicat test method is analyzed. A method of acoustic emission is also used and acoustic signals recorded in cement paste during hydration are presented.

Key words: Cement hydration, setting time, ultrasonic measurements, acoustic emission.

1. Introduction

In this paper an ultrasonic method of testing the cement hydration is presented. The hydration of cement is a process which enables concrete to harden. In the first stages of hydration concrete transforms from liquid to solid state. The transformation from liquid to solid state is characterized with two points in hydration time: initial and final setting. The initial set is important because it provides an estimate when concrete has reached the point of stiffening to such an extent that it can no longer form a monolitic element with concrete poured on top of it. The time at which final set occurs is important since it provides an estimate when the development of concrete strength and stiffness starts. This is why it is important to have a method to determine setting times.

When setting time is measured, different methods are used for cement and concrete. Setting time of cement is measured using the Vicat needle test and setting time of concrete is measured using the Proctor resistance test. Setting times in both methods are based on the penetration resistance of the tested material. There are several disadvantages with this kind of testing: penetration resistance of concrete is not measured directly on concrete material itself but on mortar obtained by sieving concrete, measurements are limited to discrete points in time, there is no clear connection between material properties and measuremet results.

To improve understanding of the hydration process and setting times new methods are investigated. One group of methods consists of ultrasonic measuring techniques. Different techniques have been presented by many authors [1]. Different types of ultrasonic waves are used, for example shear waves [2, 3], longitudinal waves [4, 5]. Using these methods a finer representation of the hydration process is obtained, but there are still problems left unsolved.

A different kind of ultrasonic technique, an acoustic emission, was also used for monitoring the hydration of cement and concrete [6-8]. The acoustic emission technique is a so called passive technique. It is called passive because an ultrasonic wave is not generated artificially by the wave generator. Sensors are attached to the specimen and ultrasonic waves generated by material itself are collected.

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In the next sections work done at the Faculty of Civil Engineering - University of Zagreb is presented.

2. Experimental Procedure

2.1 Measuring System

In order to monitor the hydration of cement paste a measuring system was assembled. The system consisted of two parts for performing two types of ultrasonic measurement. One type of measurement consisted of the active technique where an ultrasonic impulse was generated by a wave generator. At the same time a passive technique i.e. the acoustic emission, was used to record waves generated by the cement paste. Both types of measurement are presented in the following sections.

2.1.1 Active Ultrasonic Technique

The measuring system consists of a pulse generator which has a pulse repetiton frequency of 0.25 Hz. The puls is converted to an ultrasonic wave through the 54 kHz piezoelectric sensor. The wave is then transmitted through the material and picked up by a receiver, which is also the 54 kHz piezoelectric sensor. The pulse generator used is an ultrasonic instrument intended for the measurement of pulse velocity in concrete. The pulse generator is connected to a PC so that information about the speed of the ultrasonic wave is recorded. A wave signal from the receiver is also collected by an acquisition unit for the acoustic emission measurement. The acquisition system used is the Physical Acoustics acoustic emission 8-channel "system-on-a-board" (PCI-8) controlled through the AEwin software. The measuring system is shown in Fig. 1.

The system is capable of collecting waveforms and then the different wave parameters are calculated from the collected signals. Typical acoustic emission wave parameters are shown in Fig. 2. During testing, the threshold level was set to 30 dB.

2.1.2 Passive Technique–Acoustic Emission Measurement

The acoustic emission measuring system used is the PCI-8 unit from Physical Acoustic Corporation with pre-amplifiers which provide 40 or 60 dB gain and two wideband sensors with frequency range from 20-1000 kHz. One sensor is used as a test sensor and the other is used as a control sensor. The test sensor is placed in the mold in contact with the cement paste and the control sensor is used to recognize signals coming from the environment which could be picked up by the test sensor. Measurement set-up is shown in Fig. 3.

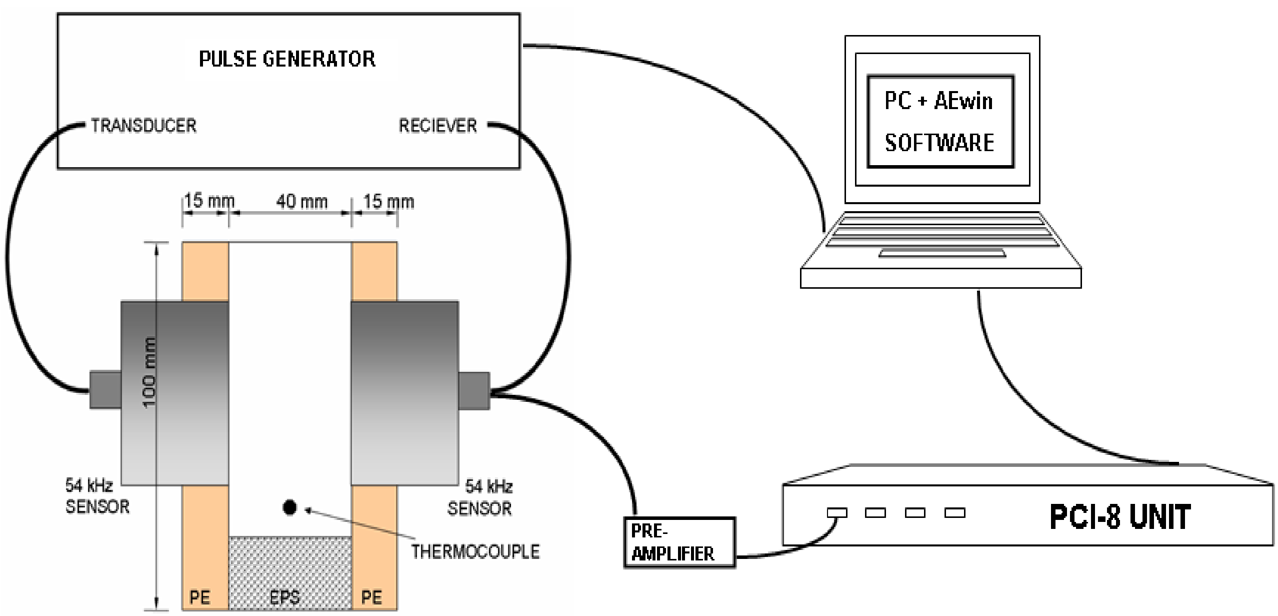


Fig. 1 Measuring system for active ultrasonic measurement.

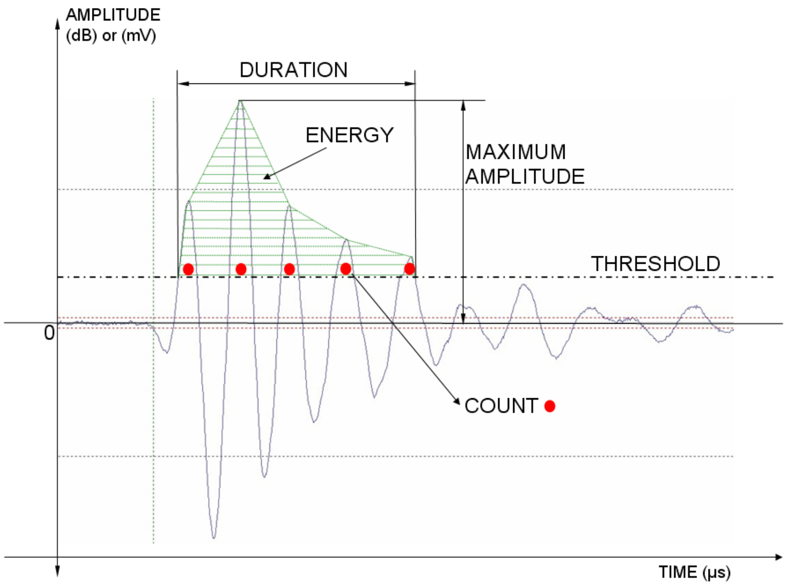


Fig. 2 Typical acoustic emission parameters.

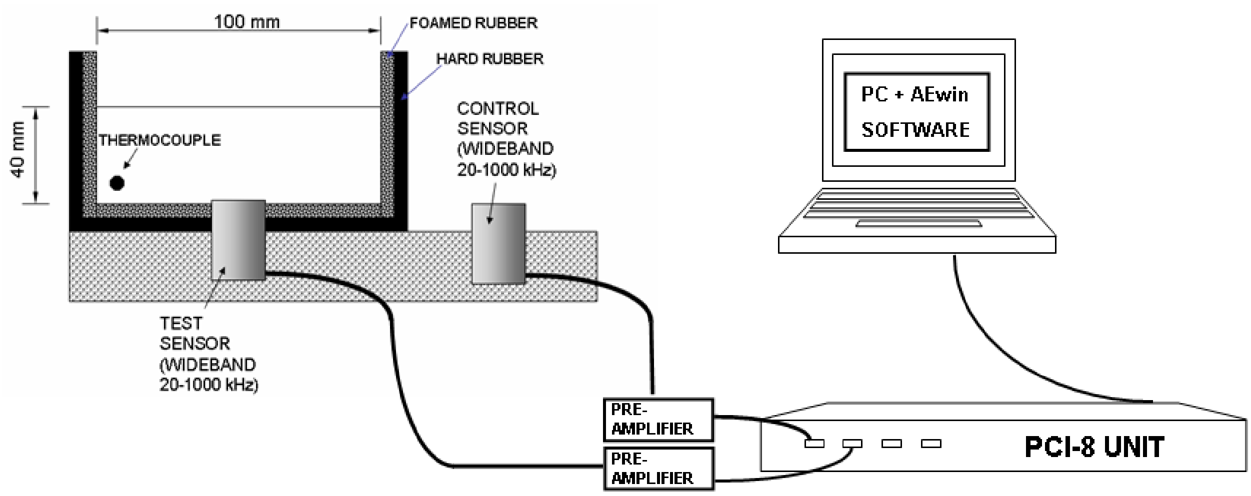


Fig. 3 Measuring system for acoustic emission measurement.

2.2 Preparation of Cement Paste

Cement used in the analysis is CEM I 42,5R. A cement paste was prepared with a water-cement ratio 0.3 by weight. The paste was mixed according to a procedure given by the standard HRN EN 196-3. Initial and final setting times of the cement paste were determined using the Vicat needle method according to the same standard. Properties of cement are shown in Table 1.

After mixing, the cement paste is placed in the molds. Two types of molds are used, one for active and the other for the passive method of testing. A mold for the active testing method is shown in Fig. 1. The mold is made of polietilen (PE) material with dimensions 100×200×40 mm. The distance between sensors was 40 mm.

A mold for the passive method consisted of hard rubber with the mold's inside overlayed with foamed rubber. Foamed rubber was placed inside of the mold in order to move together with a cement paste sample and thus reducing noise produced by movement of the specimen due to temperature change or shrinkage. Dimensions of the specimen were 100×100×40 mm. After placing, the cement paste mold was covered with a plastic sheet to reduce water evaporation from

Table 1 Properties of cement CEM I 42.5 R

Fineness Passing % (45 µm)	Blaine (m^2/kg)	Mineral composition (%)				Setting time (h) w/c ratio = 0.3	
		C_3S	C_2S	C_3A	C_4AF	Initial set	Final set
92.20	353	59.86	11.94	8.36	8.82	2.33	3.00

the specimen's surface. Temperatures of the environment and cement paste were monitored using the copper-constantan thermocouples and recorded by the Guardian instrument from German Instruments.

3. Results of Testing

3.1 Active Ultrasonic Technique

Different wave parameters are evaluated in comparison with the initial and final seting times of cement paste and shown in graphs (4a) to (4e) in Fig. 4. Comparison between changes in the ultrasonic pulse velocity and different acoustic emission wave parameters is also made and shown in Fig. 4. Fig. 4 graphs show zero time at the moment of mixing of cement and water.

In the Fig. (4a) temperature recorded in cement paste is plotted against time. Change in ultrasonic pulse velocity versus time is also plotted at the same graph. The graph shows that the temperature increase starts approximately at the time of initial setting. Speed of ultrasound at the moment of initial set is 1180 m/s and at the moment of final set 1440 m/s. It can be noticed from the graph that the increase in the ultrasonic pulse velocity starts before initial setting time as already determined by the Vicat needle test. The ultrasonic pulse velocity started to increase 1 hour and 8 minutes from the time of mixing of cement and water.

In Fig. (4b) the energy of the ultrasonic pulse is plotted against time. Energy of the pulse is calculated as the surface under the rectified voltage signal over the duration of AE hit. This is a property of the signal dependant on the amplitude and duration. In the same graph the ultrasonic pulse velocity and setting times are also plotted. Increase of the signal energy starts at approximately the same moment as the increase of the ultrasonic pulse velocity and shows the same trend.

In Fig. (4c) the number of counts is plotted against time. Increase in the number of counts starts at approximately the same moment as the increase of the ultrasonic pulse velocity and shows steep increase at the time around final setting time.

In Fig. (4d) the duration of the signal over the threshold level is plotted against time. Increase of the signal duration starts at approximately the same moment as the increase of the ultrasonic pulse velocity and shows similar trend. The graph shows that the steep increase in the signal duration appears around final setting time.

In Fig. (4e) the peak frequency of the signal, obtained by the Fast Fourier transform, is plotted against time. The graph shows that there is a leap in the frequency at the moment around initial setting time.

3.2 Passive Ultrasonic Technique

During the passive ultrasonic measurement, an accoustic emission activity in the cement paste sample was recorded and presented in Fig. 5. In Fig. (5a) the paste temperature is plotted against time. The same graph shows a change in energy of the ultrasonic signal. It can be seen that the greatest acoustic emission is recorded at the time of final setting. Fig. (5b) shows the signal amplitude. It recorded that the amplitude is largest at the time of final setting.

4. Conclusion

A system for monitoring cement hydration is presented in the paper. Two ultrasonic measuring techniques, active and passive, are used. Properties of ultrasonic waves are recorded and analyzed with respect to the setting and hardening processes in cement paste. From the obtained results it can be seen that the active ultrasonic technique presented here gives a large number of parameters which can be used

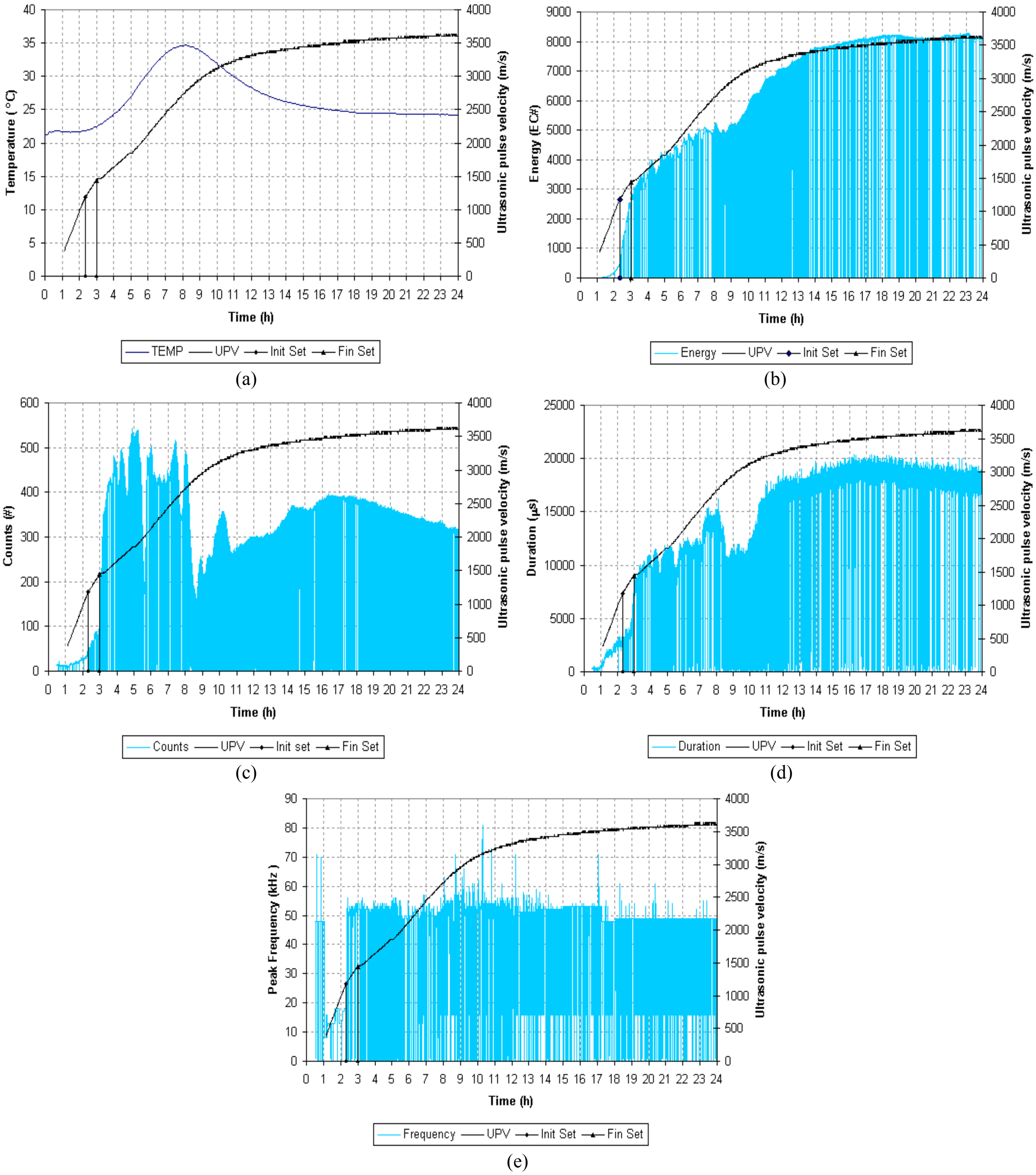
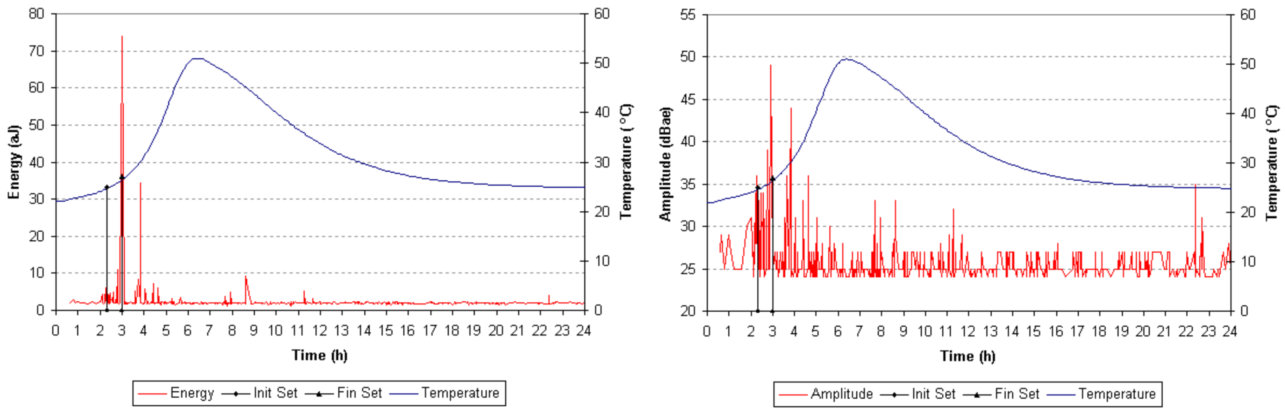


Fig. 4 Correlation between setting time, ultrasonic pulse velocity and acoustic emission parameters.

to indicate some processes in cement paste during hydration. Typical acoustic emission parameters like counts, duration and energy of the ultrasonic signal can be used to identify changes in the structure of material.

The passive ultrasonic technique presented here can be refered as the pure acoustic emission technique. From signals collected during the hydration of cement paste it can be seen that the greatest emission is generated during the setting process.



(a) (b)

Fig. 5 Correlation between setting time, temperature and acoustic emission parameters.

In order to get more insight in connection between setting, hardening and the ultrasonic wave parameters further experimental work will follow. Results presented in this paper will serve as a good starting point for future work.

Acknowledgements

This research was performed within scientific project "From Nano- to Macro-Structure of Concrete", 082-0822161-2990, funded by the Croatian Ministry of Education, Science and Sport.

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