TESTING METHOD FOR QUICK DETERMINATION OF FRESH CONCRETE SAMPLE QUALITY BY MEASUREMENTS AT ELASTIC PROPERTIES OF HYDRATING CEMENT PASTE

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ABSTRACT: Testing methods for measuring the properties of a fresh concrete are of special interest because it is possible, on the basis of its results, to predict the properties of concrete prior to its placement. The presented method is based on the ultrasonic shear waves reflection from the upper layer of the hardening cement paste. The acoustic attenuation, of the normal incidence and the reflected waves, correlates with the shear modulus of elasticity and the viscosity.

INTRODUCTION

Experimental setup for the cement slurries hydration time measurements by the ultrasonic shear waves is aimed at the industrial plant laboratories for the cement works. It can be applied for measurements at monitoring sites (during the concrete preparation), determination of the admixture characteristics and its influence on the concrete properties. Such measurements enable planing of production and fast development of the special concrete [1]. In comparison with the standard testing methods its advantage is determination of concrete properties in its early phase of formation.

- Application of acoustic emission method to determine the characteristics of the different types of cement mixtures for production of the special concrete, has accelerated the development of the special concrete. Mechanical properties of age concrete can be predicted by measuring the acoustic impedance of fresh cement slurry. Such indirect way for determination of the age concrete mechanical properties, could be applied by the contractor on delivery of the fresh concrete prior to its placement.

- From proper prepared sample, the concrete properties in the industrial production could be defined in a few hours. This is very important in the case when the big quantities of cement paste are to be monitored and its quality is not consistent.

COURSE OF CEMENT MORTAR HYDRATION TIME MEASUREMENTS

We have researched cement slurry hydration time measurements for the special concrete mixture with the experimental setup for measurement at elasticity changing of the cement paste sample by the ultrasonic shear waves. The frequency of ultrasonic transmitter/receiver (2.5 MHz) has been determined according to the technical characteristics of the selected experimental setup and the average sand grain size. The sand was used as aggregate in the cement mortar.

The results of the cement paste hydration kinetics measurements were used to determine the most suitable conditions in the ultrasonic measuring chamber. The factors of these conditions are hydration temperature and relative humidity. The humidity Standard DIN 50 008 was used as a base for determination of relative humidity.

Standard cement mortar sample was prepared according to the Standard CSA A5, ASTM C 190 [2]. This Standard is aimed at the cement mortar test tubes for the determination of the cement properties using the standard mechanical testing methods. As a filler for production of the cement mortar (v/c = 1/2, c/p = 1/3) is usually selected the siliceous sand with average grain size from 0,15 mm to 0,50 mm.

Preliminary cement paste hydration kinetics measurements by the ultrasonic shear waves were performed on the prototype measuring system with the frequency 20 MHz. Using the results of the preliminary measurements we have set down the guidelines for starting and using such experimental setups.

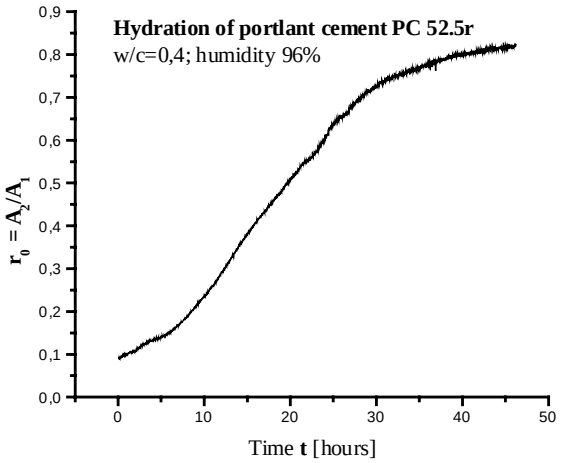

Figure 1: Portland cement paste modulus elasticity changes during the various stages of the hydration process.

The course of hydration kinetics measurements has five successive steps. The experimental setup has to be connected to a power source at least half an hour before starting the measurements, as it has to warm on its working temperature. Within this period of time the temperature in a measuring chamber would be stabilized. After the successful setting of the measuring devices, we can start with the cement paste sample preparation. The thin layer of a specimen, about 5 mm, is to be put on the top surface of the probe on the ultrasonic buffer rod. The surface has to be degreased, clean and dry. The specimen was left 48 hours in the measuring chamber to harden through a hydration process. After that the specimen can be unsticked from the probe and examined. It has not to be slurry but it has not to be dusty or crumbly as well, its sides has to be smooth. This is a basic test for verification of the cement

hydration measurements validity. The complete examination of the data validity can be performed only by the image processing of the results obtained [Figure 1].

The microscopic observation is the most reliable way for determination of the concrete specimen quality, however it requires a lot of time and preparation. For such observation the specimen has to be already hardened. Before the microscopic observation, the polishing and then etching have to be performed. The concrete quality can be verified through the microscope observation of the grain size and its distribution in a specimen.

APPLICATION OF THE ULTRASONIC WAVES IN THE GRAIN SIZE ESTIMATION

When the ultrasonic wave propagates through the material it is partially reflected from its grains. The reflected wave in comparison to normal incidence significantly changes its characteristics. Such reflected ultrasound contains also the average grain size data. However, this data can not be measured directly, as the reflected ultrasound depends on numerous factors: the frequency of the ultrasonic waves, irregularity of the specific grain shape and its distribution in material. The reflected ultrasound (backscattered signal) itself, consists of interfering multiple randomly distributed reflected sounds (backscattered echoes) with random amplitudes. Thereupon, it is difficult to evaluate accurate grain size with conventional signal processing techniques.

Many of the prior studies examined correlation between ultrasonic attenuation and material characteristics. This correlation is based on the fact that the amplitude, of the transmitted ultrasound wave, decays with distance. The sample for the amplitude decay measurements consists of two parallel surfaces, and the reflection from the back surface is measured. One specimen is of known grain size and the other is a specimen of unknown grain size. The decay of amplitude with distance results in ultrasonic attenuation subject to grain size. Consequently, the grain size estimation is obtained by comparing the results of the ultrasonic attenuation measurements for both specimens, while maintaining the uniform test conditions [3]. Such attenuation measurement techniques for estimation of the integrated grain size are relatively simple, but they are limited by surface irregularities and coupling.

The more accurate determination of the grain size can be made by measuring the attenuation of the backscattered echoes from the grain boundaries, when ultrasonic wave travel in solids. It was demonstrated that the attenuation of the backscattered echoes with the depth, while the ultrasonic wave is propagating in solids, is in relation with the average grain size. The accuracy of average grain size estimation has been dependent on accuracy of the backscattered echoes attenuation measurements. To find more accurate ways to determine the amplitude of the backscattered echoes related to depth, the researchers have brought into use various averaging techniques. These techniques can not be applied in the cases when determination of the average grain size on a specific place of sample is requested, and if the attenuation of the backscattered echoes is not big enough. The method based on spectral analysis of randomly distributed ultrasound scatterers from the grains in material, is applicable in all these cases [4].

Determination of the grain size in polycrystalline and nonhomogeneous materials by ultrasound backscattered echoes from the grain boundaries has important application in practice. Average grain size in material determines its quality and homogeneity. It is also important in estimation of the material quality with non-destructive testing methods. Thereupon, the ability to estimate an average grain size in the fresh concrete sample by ultrasound has drawn our attention. It would enable us to determine the quality of the prepared fresh cement mortar sample and to examine weather the specimen fits tight to probe from the very beginning of the hydration kinetics measurements by ultrasonic shear waves.

SPECTRUM OF RANDOMLY DISTRIBUTED SCATTERERS

Ultrasonic scatterer is composed of N scatterers [5]. For a one-dimensional random point scatterer model, the amplitude a_i and time τ_i, for individual random scatterer, are uncorrelated variables (1).

$$g(t) = \sum_{i=1}^{N} a_i \delta(t - \tau_i) \quad (1)$$

As the system is band limited, the received scatterer signal is the convolution of the scatterer function g(t) and the system impulse response h(t). In symbolic form this can be expressed with Equation (2).

$$r(t) = g(t) \otimes h(t) \quad (2)$$

In Equation (1) the variable τ_i is the time between the reference point and the i-th scatterer and is called the time of flight. The time of flight can be expressed as summation of the individual times s_k , where s_k is the traversing time for the transmitted wave between the (k-1)th and kth scatterer (3).

$$\tau_i = \sum_{k=1}^{i} s_k \quad i = 1, 2, 3, ..., N \quad (3)$$

Because τ_i is the summation of statistically independent periods from s_1 to s_i, its probability density function (pdf) can be expressed as the convolution of i individual pdf functions (4).

$$f_{\tau_i}(\tau_i) = f_{s_1}(s_1) \otimes f_{s_2}(s_2) \otimes ... \otimes f_{s_i}(s_i) \quad (4)$$

According to probability theory the characteristics function $\Phi(\omega)$ of a random variable is the complex conjugate of the Fourier transform from its probability density function $f_{\tau i}(\tau_i)$ [6]. Therefore, the characteristic function of the time s_k is defined with Equation (5), where $\omega=2\pi f$.

$$\Phi_{s_k}(-\omega) = E\{\exp(-j\omega s_k)\} = F\{f_{s_k}(s_k)\} \quad (5)$$

If we assume that the time increments between individual scatterers s_1 to s_i are statistically independent and identically distributed (iid), the characteristic function of τ_i can be expressed with Equation (6).

$$\Phi_{\tau_i}(-\omega) = E\{\exp(-j\omega s_1)\}.E\{\exp(-j\omega s_2)\} ... E\{\exp(-j\omega s_i)\} = \Phi_s^i(-\omega) \quad (6)$$

By taking the Fourier transform of Equation (1), the scatterer spectrum can be obtained (7)

$$G(\omega) = \sum_{i=1}^{N} F\{a_i \delta(t - \tau_i)\} = \sum_{i=1}^{N} a_i \exp(-j\omega\tau_i) \quad (7)$$

Since a_i and s_i are assumed to be uncorrelated and individually iid, from Equations (6) and (7) the statistical scatterer spectrum becomes (8).

$$E\{G(\omega)\} = \sum_{i=1}^{N} E\{a_i \exp(-j\omega\tau_i)\} = \mu_A \sum_{i=1}^{N} \Phi_s^i(-\omega) = \mu_A \frac{\left[1 - \Phi_s^N(-\omega)\right]\Phi_s(-\omega)}{1 - \Phi_s(-\omega)} \quad (8)$$

Coefficient μ_A is the mean of a_i and $\Phi_s(\omega)$ is characteristic function of s_k. Where s_k is individual time for kth scatterer to traverse. Therefore, the statistical scatterer power spectrum can be obtained from Equation (8).

$$\left|E\{G(\omega)\}\right|^2 = \mu_A^2 \frac{\left|1 - \Phi_s^N(-\omega)\right|^2 \left|\Phi_s(-\omega)\right|^2}{1 - \left[\Phi_s(-\omega)\Phi_s^*(-\omega)\right] + \left|\Phi_s(-\omega)\right|^2} \quad (9)$$

In Equation (9), a complex conjugate is denoted with *. Thereupon, for any scatterer distribution for which the characteristic function $\Phi_{\tau i}(-\omega)$ exists, the statistical scatterer power spectrum can be obtained using Equation (9).

AVERAGE GRAIN SIZE DETERMINATION

If the scattered distribution s is assumed to be Gaussian distributed with mean μ and standard deviation δ, then its characteristic function and the statistical scatterer power spectrum can be obtained [5]. This power spectrum exhibits nearly periodic peaks. The frequency spacing between these spectral peaks and the average scatterer spacing have an inverse relationship.

As standard deviation δ increases, the peaks of the statistical scatterer power spectrum will widen and the magnitude of the higher order harmonics will reduce more rapidly with frequency.

If some assumptions are satisfied, and the grain size has Gaussian distribution with mean grain size μ_l and standard deviation δ_l the statistical grain power spectrum can be obtained. It will be a function of frequency, average grain size μ_l , standard deviation δ_l and the number of grains N. For infinitesimal values of standard deviation, a special case occurs, which results in periodic pulses with frequency spacing Δf, which is related to the average grain size (10). In this Equation c is the ultrasonic propagation velocity in the sample.

$$\Delta f = \frac{c}{2\mu_l} \qquad (10)$$

The theoretical results have shown that the statistical scatterer power spectrum is uniquely determined by the scatterer pdf (probability density function). It is also shown that the statistical scatterer power spectrum can be used for various signal processing. One of its application is average grain size estimation. With the randomly distributed scatterer model researchers have shown the correlation between the statistical grain power spectrum and average grain size [4, 5, 7]. Such theoretical model we have developed in our laboratory for performing the quality measurements of the standard cement paste samples. This theoretical results have been examined in practice and proved as applicable for average grain size estimation by grain power spectrum [8, 9, 10].

CONCLUSION

The ultrasonic cement paste and cement mortar hydration kinetics measurements are based on the reflection coefficient measurement. Ultrasonic waves with suitable length are generated by the transducer and travel through the buffer rod to the sample to be measured.

A specimen for the hydration time measurement by the ultrasonic shear waves have to be prepared and set down onto the top of the buffer rod. The first examination of the specimen preparation successfulness and results of its hydration kinetics measurement can be partly verified not earlier than after 48 hours. With additional determination of the statistical scatterer power spectrum it would be possible to determine sample preparation quality and weather it fits tightly to ultrasonic probe or not.

For the accurate estimation of the cement mortar sample preparation quality we should preliminary collect the adequate comparable data on elastic properties of the previously used cement and data on other cement mortar compounds used. Such comparable data can be used for general application. For instance, in the case of hydration kinetics measurements by HF ultrasonic shear waves for verification of its quality, or in industrial production of concrete for keeping its quality consistent.

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