

Nondestructive Monitoring of Setting and Hardening of Portland Cement Mortar with Sonic Methods

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Abstract

The setting and hardening process of concrete is considered to be the most critical time period during the life of a concrete structure. Previous research has been conducted on an ultrasonic wave reflection method that utilizes a steel plate embedded in the concrete to measure the reflection loss of shear waves at the steel-concrete interface. The reflection loss has been shown to have a linear relationship to compressive strength at early ages. A procedure for strength prediction based on measured loss has been developed.

The presented investigations continue this research by examining the fundamental relationship between the reflection loss, measured with shear waves, and the hydration kinetics of Portland cement mortar, represented by setting time, dynamic elastic moduli, compressive strength and degree of hydration. Dynamic elastic moduli are measured by fundamental resonant frequency and ultrasonic pulse velocity using compression and shear waves. Degree of hydration is determined by thermogravimetric analysis and adiabatic heat release. The water/cement ratio was varied for the tested mixture composition.

The results presented herein show that compressive strength, dynamic shear modulus and degree of hydration have a linear relationship to the reflection loss for the tested mortars at early ages. This trend indicates that test methods based on measuring dynamic shear moduli have the ability to measure mechanical properties microstructural changes that determine early age properties of hydrating cementitious materials. The presented results validate and verify the previously developed strength prediction procedure based on the shear wave reflection method.

Introduction

The nondestructive, in-situ testing of early-age concrete properties is a crucial tool for the progress of many construction projects in the building sector. The application of such techniques can establish the earliest possible form removal from concrete construction elements, thereby opening highways to traffic, releasing prestress from steel reinforcement, or applying post-tensioning with greatest efficiency.

A nondestructive, ultrasonic technique, which measures the reflection loss of ultrasonic shear waves from the concrete surface, was developed at the Center

for Advanced Cement-Based Materials at Northwestern University [1]. The focus of this research is to develop a nondestructive field sensor for in-situ monitoring of the setting, hardening, and strength gain of cementitious materials.

The research conducted so far has shown that the wave reflection method is sensitive to and can measure the hydration progress of cementitious materials. The differences in the hydration rate caused by factors such as retarding or accelerating admixtures can be detected reliably [1]. The experiments have also indicated that the method is able to follow the compressive strength gain of the test material [2], [3].

The investigations presented in this paper are aimed at studying the fundamental relationship among evolving microstructure, mechanical properties, and ultrasonic wave reflection measurements. The reflection loss measured with shear waves can theoretically be related to shear modulus. The development of shear modulus with time is related to how the microstructure of hydrating cement evolves as a result of curing. Experiments are designed to elicit this fundamental understanding. The hydration behaviour of different cement mortar mixtures will be investigated by the wave reflection method and alternate test methods. The comparison of the test results will yield the material parameters that govern the measured reflection loss.

Wave Reflection Method

The wave reflection method monitors the reflection loss of ultrasonic shear waves at an interface between a steel plate and a cementitious material over time. The amount of the lost wave energy depends on the reflection coefficient, which in turn is a function of the acoustical properties of the materials that form the interface.

A schematic of the experimental technique is shown in Fig. 1. A steel plate is embedded in the concrete. A transducer with a center frequency of 2.25 MHz, which is attached to the steel plate, transmits a shear wave pulse into the steel. The pulse undergoes a multiple reflection process, which is shown in Fig. 1.

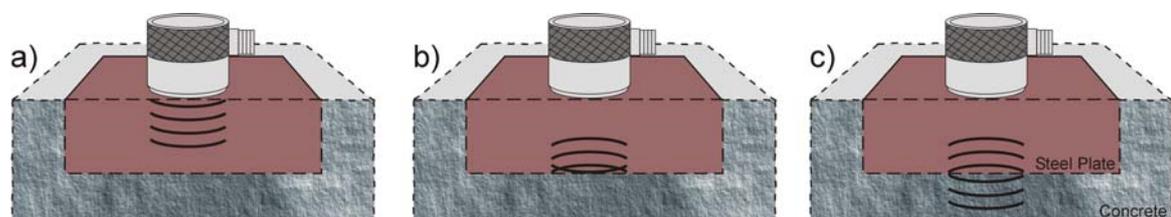


Fig. 1. Schematic of multiple reflection process

The transducer transmits a pulse into the steel plate (Fig. 1a). When the concrete is in liquid state the pulse is entirely reflected at the steel-concrete interface, since shear waves do not propagate in liquid materials (Fig 1b). With progressing hydration, the microstructure built up by the hydration products allows the shear waves to propagate into the concrete: the pulse is partially reflected and

transmitted at the steel-concrete interface (Fig 1c). The reflections are received by the transducer and used for the calculation of the reflection loss. The pulse transmitted into the concrete attenuates and is not further evaluated. The described reflection process repeats several times until the transducer transmits a new pulse.

After transforming the received signals from time domain into frequency domain, the reflection loss can be calculated from the difference between the amplitudes of the first and second reflections. In this paper the reflection loss will be expressed in decibel and describes the reduction in amplitude of the traveling shear wave due to transmission losses at the steel-concrete interface. The complete numerical procedure for calculation of the reflection loss can be found in [2].

The experimental setup, which is used for the wave reflection test is given in Fig. 2. It basically consists of a laptop computer, a pulser/receiver, a transducer and, a steel plate. The transducer, which generates the ultrasonic waves, is connected to the computer via the pulser/receiver. This unit excites the transducer and transmits the information of the received reflections from the transducer to the computer. The setup shown in Fig. 2 is capable to measure the reflection coefficient at two separate channels. Consequently, by using two transducers, the reflection loss can be measured simultaneously at two different points at the specimen or structure.

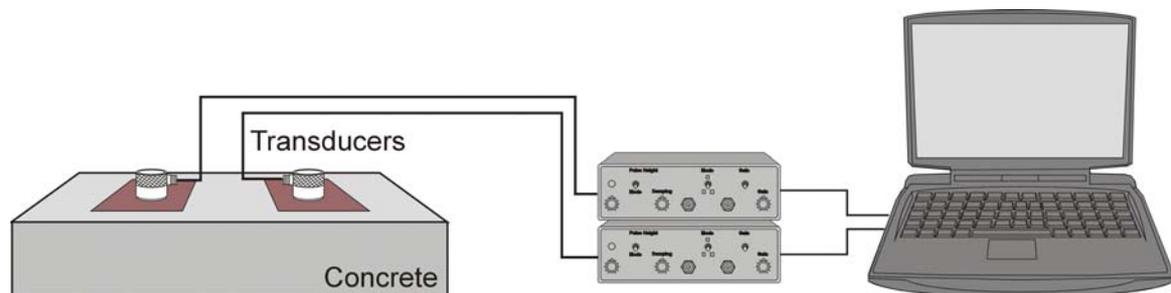


Fig. 2. Experimental setup

Experimental Program

The experimental study was conducted on cement mortars containing Portland cement type I (after ASTM C-150) and silica sand as fine aggregates. The cement mortar was tested in three different water-cement ratios: 0.35, 0.5, 0.6. The mixture composition of the mortars is given in Table 1.

Table 1. Mixture proportions by weight of cement for tested mortars

mixture	cement	water	sand
A	1	0.35	2
B	1	0.50	2
C	1	0.60	2

To determine the hydration behaviour of the mortar mixtures the reflection loss and three alternate material parameters were measured: compressive strength, dynamic shear modulus and degree of hydration. The tested materials were cured at a constant temperature of 25°C throughout the duration of the experiments. The conducted experiments and their results are described in the following paragraphs.

Reflection Loss

The development of the reflection loss throughout the setting and hardening of the three mortar mixtures was measured as described previously. The results are given in Fig. 3.

Compressive Strength

The compressive strength development was determined according to the ASTM-Standard C 109. Mortar cubes with the size of 50x50 mm were used for the compression tests. The cubes were cured in a temperature controlled water bath at 25°C. Dependent on the w/c-ratio the tests were started between four and six hours after casting. The evolution of the compressive strength of the three mortar mixtures for the first 72 hours is shown in Fig. 4.

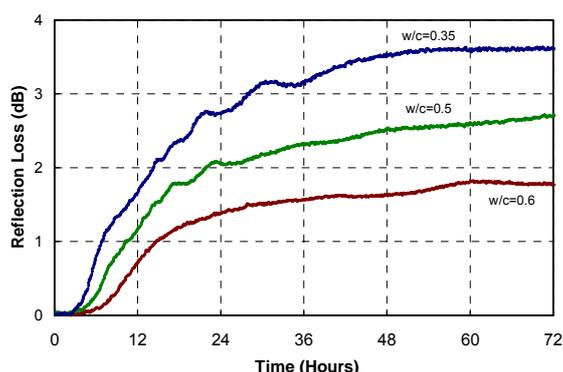


Fig. 3. Development of reflection loss

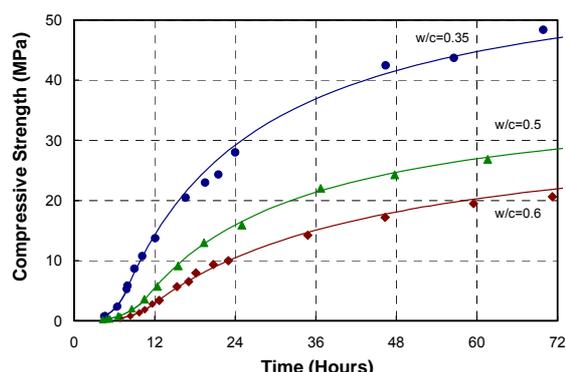


Fig. 4. Development of compressive strength

Dynamic Shear Modulus

The dynamic shear modulus was tested by using the forced resonant frequency method which is described in ASTM-Standard C-215. The dynamic shear modulus (or dynamic modulus of rigidity) was calculated from the fundamental torsional resonant frequency of mortar prisms of the size 210x60x50 mm. The fundamental torsional resonant frequency of the prisms was measured by forcing the specimen to vibrate in torsional mode and identifying the frequency with the highest amplitude. The measured dynamic shear moduli are presented in Fig. 5.

Degree of Hydration

The degree of hydration was determined by thermogravimetric analysis (TGA). This test allows the determination of the amount of non-evaporable water per gram of original cement of a cement paste sample. The samples were heated up

to a temperature of 900°C according to a defined temperature profile and the weight loss due to the evaporation of the free and the decomposition of the chemically combined water was measured. The temperature was first increased to 105°C, held at that value for 2 hours and then increased further up to 900°C. The heating rate was 10°C/min. The samples were heated in a steady flow of dry, CO₂-free nitrogen. From the difference of the sample weight at 105°C and 900°C the amount of non-evaporable water was determined (Eq. 1). The amount of non-evaporable water corresponds to the fraction of water that is chemically combined by the hydration process. By relating the amount of non-evaporable water at a certain time *t* to that for complete hydration, the degree of hydration can be calculated (Eq. 2). The results for the degree of hydration development in the first 72 hours is given in Fig. 6.

$$\frac{W_n}{C} = \frac{W_{105^\circ\text{C}}}{W_{900^\circ\text{C}}} (1 - L) - 1 \quad (1)$$

$$\alpha(t) = \frac{\frac{W_n(t)}{C}}{\frac{W_n}{C_{\text{complete}}}} \quad (2)$$

$\frac{W_n}{C}$	non-evaporable water per gram original cement	$\frac{W_n}{C_{\text{complete}}}$	non-evaporable water for complete hydration
$W_{105^\circ\text{C}}$	sample weight after heating for 2 h at 105°C	$\alpha(t)$	degree of hydration at time <i>t</i>
$W_{900^\circ\text{C}}$	sample weight after heating at 900°C	<i>L</i>	loss on ignition of original cement

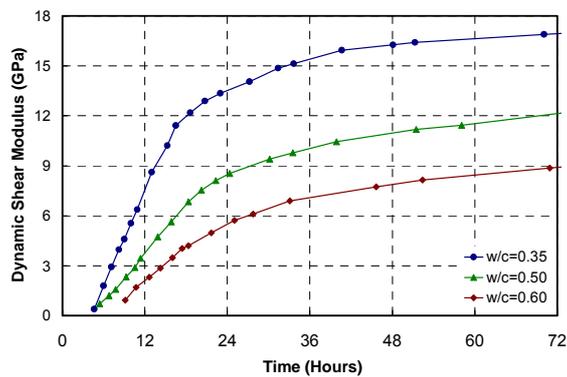


Fig. 5. Dynamic shear moduli determined from fundamental torsional resonant frequency

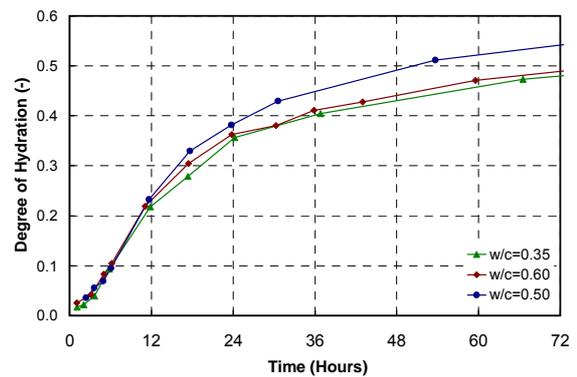
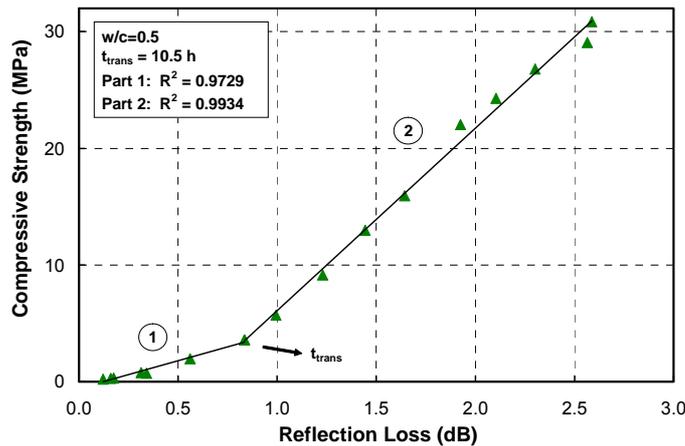


Fig. 6. Degree of hydration determined from TG measurements

Relationship between Reflection Loss and Compressive Strength

The relationship between compressive strength and reflection loss was already established previously in [2] and [3]. It was found that both parameters are linear related for mortars and concretes with varying composition and curing conditions in a time range of up to four days. The linearity of this relationship could be reproduced for the mortars tested in this study. Due to the very early start time of the compressive strength tests (around initial set) an additional feature of the strength-reflection loss relationship (*S*–*R_L* relationship) could be identified.

As shown in Fig. 7, the S–R_L relationship exhibits a strong bilinear pattern, dividing the relationship into two parts. The first part at very early ages has a clearly lower slope compared to the second part of the relationship at later ages. The slope changes at a certain time, which is 10.5 hours for the shown mortar with w/c = 0.5. It was observed, that the time of transition (t_{trans}) between the two slopes changes with the kinetics of the strength gain. The low w/c-ratio (0.35), which corresponds to a faster increase in strength, shows an earlier transition time, whereas the high w/r-ratio (0.6) shows a later transition time, respectively.



It is assumed that the bilinear behaviour of the S–R_L relationship is attributed to changes in the growth characteristic of both parameters: reflection loss as well as compressive strength.

Fig. 7. Relationship between reflection loss and compressive strength for mortar with w/c=0.5

Relationship between Reflection Loss and Dynamic Shear Modulus

The relationship between the dynamic shear modulus and reflection loss is an essential part for understanding how the wave reflection measurements are related to fundamental material parameters. The reflection loss is an expression of the reflection coefficient at the steel-mortar interface. The reflection coefficient measured with shear waves can theoretically be related to the shear wave velocity of the tested mortar (Eq. 3 and 4). By knowing the acoustic impedance of the used steel plate the dynamic shear modulus of the mortar can be calculated (Eq. 5). It will be analyzed in the following how the dynamic shear modulus calculated from reflection loss G_r is related to the dynamic shear modulus measured by fundamental torsional resonant frequency G_{tors} (Eq. 6).

$$r = \frac{Z_{mortar} - Z_{steel}}{Z_{mortar} + Z_{steel}} \quad (3)$$

$$Z_{mortar} = \rho_m \cdot v_{m, shear} \quad (4)$$

$$G_r = v_{m, shear}^2 \cdot \rho_m \quad (5)$$

$$G_{tors} = B \cdot W \cdot (n'')^2 \quad (6)$$

r reflection coefficient
 Z acoustic impedance
 $v_{m, shear}$ shear wave velocity in mortar
 ρ_m density of mortar

n'' torsional resonant frequency
 W weight
 B shape + size factor

The comparison of G_r and G_{tors} in time for the mortar with $w/c = 0.5$ is shown in Fig. 8. First, it can be noted that both curves have a similar shape. The differences in the absolute values of G_r and G_{tors} can be explained by the following theory: The wave reflection loss measures the properties of the material located next to the steel plate. It is assumed that only the cement paste properties influence the reflection loss. In contrast to that, the resonant frequency method measures bulk properties of the tested mortar. Based on that theory, G_r represents the properties of the cement paste only whereas G_{tors} is a function of the bulk properties of the mortar. The relationship between G_r and G_{tors} is given in Fig. 9. The linear trend indicates that the reflection loss strongly depends on the evolution of the dynamic shear modulus and consequently measures an important mechanical material property.

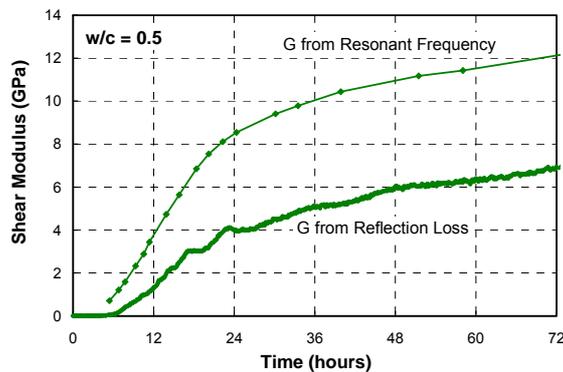


Fig. 8. Dynamic shear moduli calculated from reflection loss and torsional resonant frequency

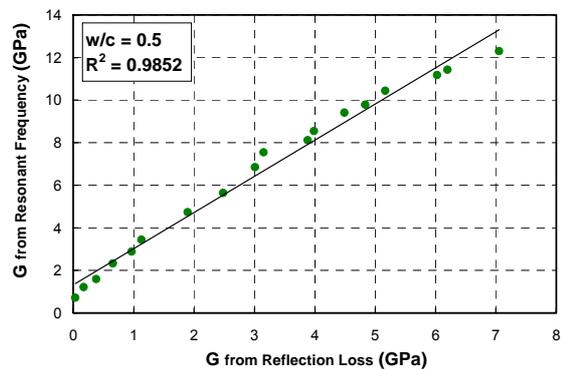


Fig. 9. Relationship between G_r and G_{tors}

Relationship between Reflection Loss and Degree of Hydration

The degree of hydration is one of the most fundamental material parameters of a Portland cement mortar. It describes the progress of the hydration reaction and is a governing parameter for many mechanical mortar and concrete properties.

The development of the degree of hydration and the reflection loss in time for the mortar with $w/c=0.35$ is given in Fig. 10. It can be seen from the figure that both quantities have a very similar trend over the time period of ca. 80 hours. The relationship between degree of hydration and reflection loss is given in Fig. 11. The presented data show a very strong linear trend over the entire period of time that is plotted. The same linear trend exists for the other two mortars tested in this study. It was also found that the slope of the relationship changes with w/c -ratio, where a low w/c -ratio corresponds to a high slope.

The degree of hydration was calculated from the amount of the non-evaporable water (Eq. 1 and 2) in the cement paste. This parameter in turn yields information about microstructural parameters of the cement paste e.g. capillary porosity, gel pores and gel volume. This analogy underlines the great potential of the wave reflection method to measure the evolution of fundamental material properties.

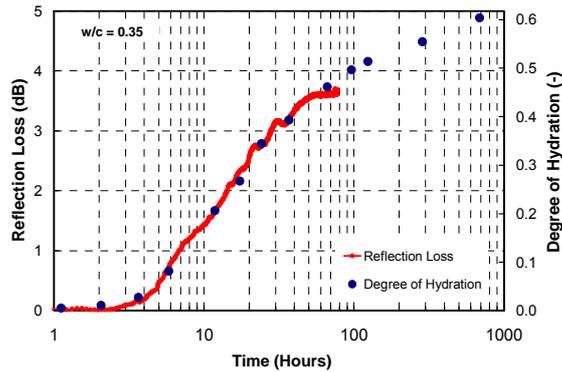


Fig. 10. Development of degree of hydration and reflection loss in time

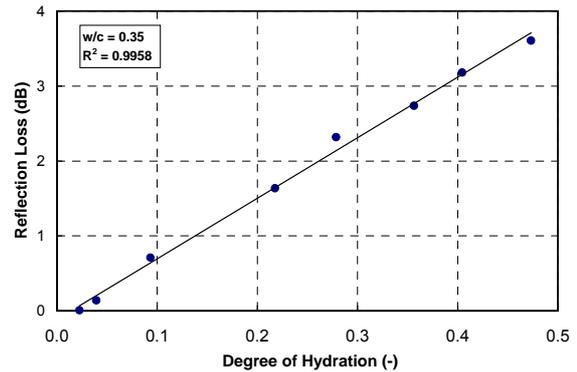


Fig. 11. Relationship between degree of hydration and reflection loss

Conclusions

From investigations presented in this paper the following conclusions can be drawn:

1. The relationship between reflection loss and compressive strength has a bilinear character for the tested mortar mixtures at early ages (70 to 90 hours). The time of the transition point depends on the kinetics of the strength gain.
2. Dynamic shear moduli calculated from torsional resonant frequency and reflection loss are linear related at early ages. This shows that the wave reflection method is governed by the evolution of an important mechanical parameter.
3. Degree of hydration and reflection loss are linear related at early ages. This demonstrates the high sensitivity of the presented wave reflection method to changes in the microstructure of cementitious materials due to hydration.

Literature

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