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Monitoring the setting behavior of cementitious materials using one-sided ultrasonic measurements

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Abstract

Cementitious materials are transformed from a fluid to a solid state due to a chemical reaction known as hydration. These cementitious materials exhibit a continuous change in the mechanical properties with time; there is a steady increase in the stiffness after setting. An ultrasonic test setup and the data analysis procedure, which provide for continuous monitoring of the hydrating cementitious materials from a very early age, have recently been developed. The test procedure for obtaining the ultrasonic test data from cementitious material at different stages of hydration and the theoretical analysis, which allows interpreting the ultrasonic response in terms of the changes in the acoustic shear impedance of the hydrating cementitious material, are presented in this paper. Experimental test results obtained from mortar mixtures of known composition are presented. It is shown that the initial and final setting times correspond approximately with the occurrence of distinctive features in the ultrasonic response.

Keywords: Hydration; Mortar; Shear modulus; Ultrasonic shear wave; Setting time; Visco-elastic

1. Introduction

Setting, stiffening and subsequent strength gains in concrete are produced by the hydration of cement. Initially after mixing, the material is in a fluid state, which allows it to be placed in the forms. With time, the concrete mixture stiffens and becomes less workable. There is a progressive change in the state of the material where the fluid state is transformed into a solid state with continued hydration. This gradual development of rigidity is known as setting. After setting, with time, the solid stiffens and gains strength. The change in the state of the material from a fluid to a solid and the subsequent gain in strength are a result of continuous evolution of the microstructure in the cementitious phase of the material. The microstructure development of the cementitious phase is responsible for the progressive change in the mechanical properties of concrete. Monitoring changes in the mechanical properties of the cementitious phase, therefore, provides an insight into the development of the microstructure.

Currently, changes in the cementitious material after mixing are followed using different techniques, each with its own limitations and applicability. Using oscillatory shear measurements, cement paste has been shown to behave like a visco-elastic fluid, which can be characterized using loss and storage moduli [12]. With hydration, the increase in storage modulus is shown to be significantly higher than that of loss modulus, signifying a change in the microstructure such that it becomes stiffer and more solid-like [12]. The beginning of setting (or emergence of a solid phase) defines the practical limit of applicability of the oscillatory shear measurements.

Changes in stiffness produced by hydration, through setting, are currently quantified by measuring the increase in penetration resistance with time. The test procedure for the pin-penetration test has been standardized in ASTM C 403

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[1]. ASTM C 403 identifies two points, defined as initial and final setting times, based on the penetration resistance reaching values of 500 and 4000 psi, respectively. The initial and the final setting times identified by the ASTM procedure approximately represent the times when concrete is no longer workable (cannot be mixed, placed and compacted) and the time at which significant development of strength takes place, respectively [8]. While the penetration resistance method has been used to monitor the increase in stiffness through setting, it does not provide for direct evaluation of useful mechanical properties. After final setting, the properties of concrete can be obtained as a function of age using mechanical tests [2,3] or vibration-based measurements performed on standard compression test specimens [13,17].

Test techniques that allow for monitoring the development of properties of a cementitious material continuously after mixing, during setting and subsequent strength gain are currently under development. Reinhardt et al. [10] developed a test device for monitoring the through thickness ultrasonic compression wave velocity in concrete after casting. The observed change in compression wave velocity with time was used to study the development of elastic material properties of concrete with varying compositions. This technique, however, does not provide sufficient sensitivity, while the concrete is in a fluid state. A one-sided ultrasonic technique, similar to the one presented here, has been used by Valic et al. [15] to study the early hydration in cement pastes. The amplitude of the ultrasonic wave reflection at the interface between cement paste and a fused-quartz rod was shown to be sensitive to the kinetics of hydration reaction. Microwave-based techniques for monitoring the strength gain of concrete have also been proposed by other researchers [16,18]. The dielectric properties obtained from a microwave measurement, however, do not provide for direct determination of the elastic properties the cementitious material.

In this paper, a nondestructive technique, which is based on high-frequency ultrasonic measurements, is used to investigate the early age response of cement mortars after mixing. The experimental procedure consists of continuously monitoring the reflection of ultrasonic waves at the interface between the form and the hydrating cementitious material after casting [9,11]. Previous studies have shown a good correlation between the measured trends of the amplitude of the wave reflection at a steel–concrete interface and the compressive strength gain in the first 72 h [14]. In this paper, the existing test setup was modified to provide increased sensitivity to changes in the stiffness of the material in the early stages of hydration (through setting). The observed trends in the ultrasonic signals are interpreted to provide an explanation of the observed response in terms of the changes in the acoustic shear impedance of the hydrating cementitious material. The early age response of mortars with varying mixture composition was investigated in the experimental program. Finally, applications for identifying the setting time of concrete based on the observed response are developed. It is shown that the initial and final setting times obtained by the ASTM C 403 procedure approximately correspond in time with the occurrence of distinct features in the ultrasonic response.

2. Objectives

The objectives of this study are: (a) to develop suitable instrumentation and experimental procedures for continuous monitoring of a cementitious material after mixing; (b) to interpret the observed ultrasonic test response in terms of physical changes in the stiffness of concrete; and (c) to investigate the early-age response of mortars as a function of mixture composition.

3. Experimental program

The experimental program consisted of testing mortars of varying compositions and setting time characteristics. A test matrix was developed to determine the ultrasonic wave reflection response as a function of material composition. The mix compositions tested in this study are shown in Table 1. For each mortar mixture composition, penetration measurements were performed as per the requirements of ASTM C 403 [1]. Penetration tests were performed on mortar specimens in a PMMA mold, which had an inner diameter equal to 150 mm (6 in.) and height equal to 175 mm (7 in.). Ultrasonic wave reflection and temperature measurements were performed on a companion specimen of identical dimensions. Temperature was recorded using thermocouples embedded inside the center of the mortar specimen. Specimens were stored in an environmental chamber, which was maintained at 23 °C. The specimens were kept sealed by covering them with a plastic sheet.

Table 1
Mixture properties

Mix number	Name	Cement, kg/m^3	Sand, kg/m^3	Water, kg/m^3	Admixture
1	Normal	390	856.5	156	N.A.
2	Accelerator	390	856.5	156	6.75 L
3	Retard	390	856.5	156	0.9 L
4	AE	390	856.5	156	0.165 L
5	Fly ash*	312	856.5	156	78 kg
6	SF	358.8	856.5	156	SF*: 31.2 kg SP*: 1.65 L
Different water/cement ratios					
7	WC_35	390	856.5	136.5	N.A.
8	WC_45	390	856.5	175.5	N.A.
9	WC_50	390	856.5	195	N.A.

Fly ash*: Class F fly ash.
SP*: High-range water-reducing admixture.
SF*: Silica/fume.