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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.

The experimental test setup for the ultrasonic wave reflection measurements is shown in Fig. 1(a). The mortar was placed in the PMMA mold after mixing. A broad-band ultrasonic transducer with 1 MHz central frequency was attached to the outside (bottom) of the mold using a rapid setting epoxy. The transducer sends an ultrasonic signal into the PMMA when it receives an excitation from the pulser–receiver. The ultrasonic signal traverses the thickness of the mold and is received by the transducer after reflection at the PMMA–mortar interface. The reflected signal was captured and digitized by the oscilloscope at a sampling rate equal to 0.1 μs. The signal was then transferred to the computer over a GPIB interface. The time domain signal was windowed using a rectangular window, zero padded to 8192 points and transformed into frequency domain using the FFT algorithm.

3.1. Theoretical basis of reflection measurements

Reflection of an ultrasonic wave at a bi-material interface introduces a change in the amplitude of the wave, when compared with the incident wave [4,6,7]. The measurements in the test procedure introduced in this paper are based on determining these changes in the amplitude of the incident wave after reflection at a bi-material interface (Fig. 1(c)). For an incident shear wave $A(f)$, the reflected wave is given as

$$\text{reflected wave} = r(f).A(f) = \frac{Z_2 - Z_1}{Z_2 + Z_1}A(f) \tag{1}$$

where $r(f)$ is the amplitude reflection factor for an incident wave of frequency f, Z_2 and Z_1 are the acoustic impedances, respectively, of medium 1 and 2. The acoustic impedance of a material is given as

$$Z = \sqrt{\rho G} \tag{2}$$

where ρ and G are the density and the shear modulus of the material, respectively. In materials that exhibit frequency-dependent elastic material properties, the $r(f)$ varies with frequency.

The choice of PMMA as the material for the mold was based on having the ability to sensitively determine early age changes in the acoustic impedance of mortar. For determining $r(f)$ using Eq. (1), in the test setup described, medium 2 corresponds with mortar. After mixing, the acoustic impedance of mortar is usually very small in magnitude. Further, the changes in the acoustic impedance of mortar due to early hydration are also of a very small magnitude. Sensitive detection of small changes in the acoustic impedance of mortar requires proper selection of material 1, such that the ratio of the acoustic impedances of the two materials is close to 1. Further, proper choice of material for medium 1 requires that it should be non-reactive with the cement. After a thorough search, PMMA was found to suitable for medium 1. Thus, in Eq. (1), medium 1/2 refers to the PMMA/mortar. Changes in amplitude reflection factor at any frequency f, $r(f)$, are produced when the acoustic impedance of the mortar changes with respect to that of PMMA. The change in density of the mortar with age is insignificant compared with the change in its shear modulus. Thus, monitoring the amplitude reflection factor provides a convenient means for monitoring changes in the shear stiffness of the mortar.

The test procedure comprised of averaging 100 signals in the time domain. The averaging procedure allows for eliminating random noise in the signal. The mold was initially filled with water reflected wave signals from the PMMA-water interface were collected and averaged. The cementitious material was placed in the mold after emptying the water. The reflected signals were then collected every ten minutes up to 16 h after casting. The magnitude of the reflected wave signals collected at the transducer is influenced by the losses produced by the couplant (epoxy) and those arising from the transmission path (Fig. 1(b)). The self-compensating technique proposed by Achenbach et al. [5] was used to determine the value of $r(f)$ from the measured time domain signal, which is free of these losses. In this technique, the computation of $r(f)$ is performed in the frequency domain. In the frequency domain, the stress wave signals received after a single reflection at the PMMA–water and PMMA–

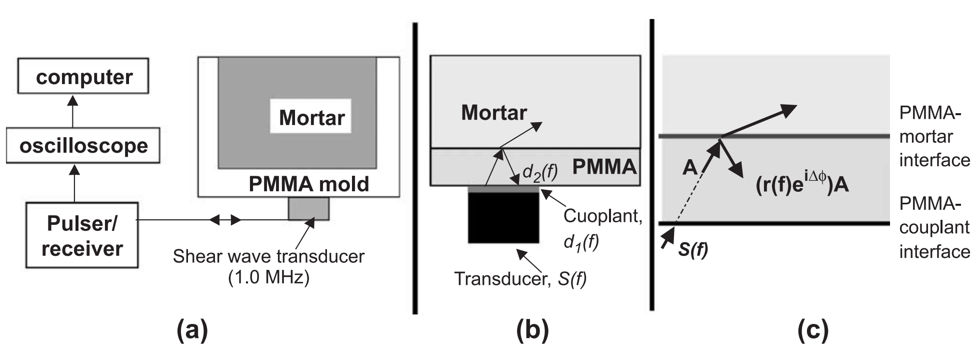

Fig. 1. (a) Schematic figure of the experimental test setup for wave reflection measurements. (b) Wave reflection at the PMMA–mortar interface showing the frequency dependent influences in the path of the stress wave. (c) Reflection at the PMMA–mortar interface.

mortar interfaces can be represented as a simple product of terms [5]

$$F_1^{\text{PMMA-water}} = s(f)d_1(f)d_2(f) \text{ and}$$
$$F_1^{\text{PMMA-mortar.}} = s(f)d_1(f)d_2(f)r(f) \quad (3)$$

where F_1 is the magnitude of FFT of the captured time domain signal, $d_1(f)$ are the geometric and material signal losses in the couplant, $d_2(f)$ the material signal losses along the signal path in the PMMA, $S(f)$ the input source function, and $r(f)$ is the amplitude reflection factor at the PMMA–mortar material interface. Water can be treated as an ideal fluid which results in a total reflection of the shear wave. The $r(f)$ for reflection at the PMMA–water interface is thus equal to 1.0. Therefore, the amplitude reflection factor, $r(f)$, can be determined by normalizing the reflected magnitude off the PMMA–mortar interface with the reflected amplitude off the PMMA water interface as shown below:

$$r(f) = F_1^{\text{PMMA-mortar}}/F_1^{\text{PMMA-water.}} \quad (4)$$

The amplitude reflection factor, $r(f)$, thus obtained using Eq. (4) defines the ratio of amplitudes of the reflected and incident signals following a reflection at the PMMA–mortar interface, which is free from the influence of losses introduced in the wave path (Fig. 1(c)).

In the procedure outlined above, changes in $r(f)$ are attributed to the changes in the acoustic impedance of the mortar. Since cement hydration is an exothermic reaction, the impact of changes in the acoustic impedance of PMMA due to temperature changes on the measured $r(f)$ was determined prior to investigating the response of mortar. Experimental measurements were performed using the PMMA mold filled with water, which was heated to identical temperatures as those recorded from the mortar samples. The variation in $r(f)$ produced by the imposed temperature change was found to be insignificant, approximately 2% of the value recorded at the ambient temperature.

4. Results and discussion

4.1. Experimental results

A typical time domain signal captured after one reflection at the PMMA–mortar interface and its corresponding representation in the frequency domain are shown in Fig. 2(a) and (b), respectively. It can be seen that there is a continuous change in the reflected waveforms in the time domain. Initially, in the first few hours, there is a very small change in reflected waveforms. However, in the next few hours, there is a rapid change in both the amplitude and the shape of the reflected waveforms collected after one reflection at the mortar interface. After approximately 10 h, a complete phase reversal is observed, i.e., the wave appears flipped when compared with that recorded in the first few hours. After the phase reversal, the wave amplitude continues to increase and approximately 16 h after mixing, the reflected wave appears like a mirror image of the wave reflection at 4 h. From Fig. 2(b), it can be seen that although there is a significant change in the shape of the reflected waveforms in the time domain, the frequency content of the reflected

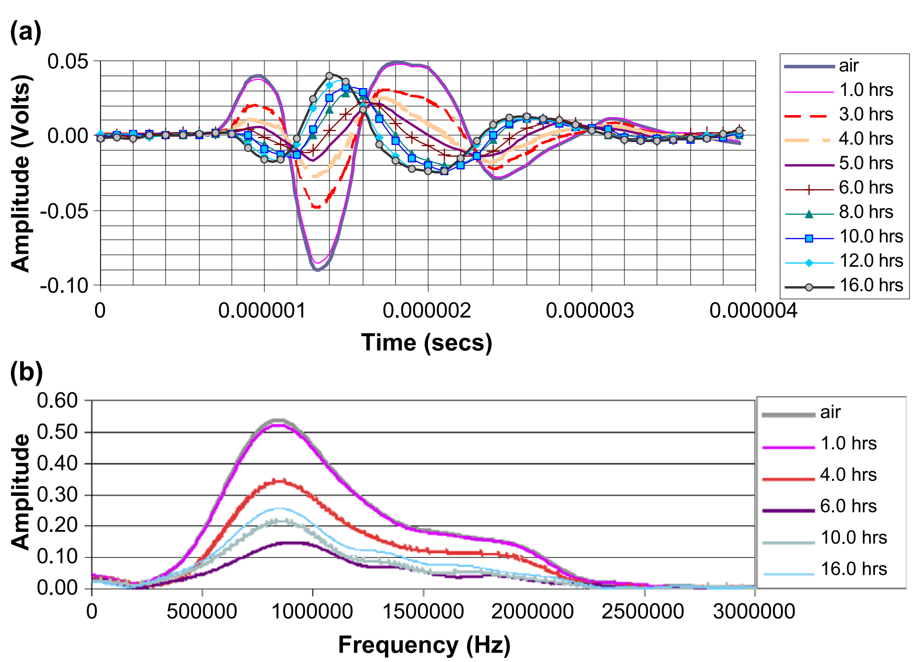

Fig. 2. (a) Time domain waveforms of the stress waves recorded after reflection at the PMMA–mortar interface. (b) FFT amplitude of the stress waves captured after one reflection at the PMMA–mortar interface.

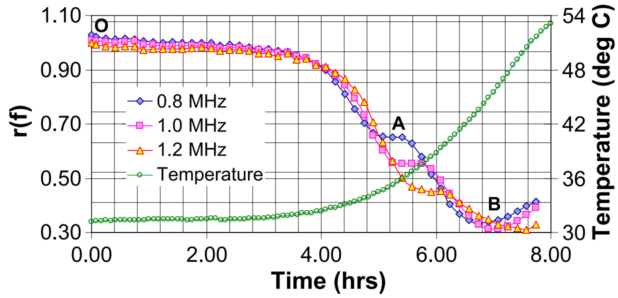

Fig. 3. The measure $r(f)$ and temperature responses as a function of time.

waves (the relative magnitudes of the different frequencies) is relatively unaltered. It is also interesting to note that there is a significant change in the reflected waveforms after the first few hours, which is discernable in both the time and the frequency domains.

The amplitude reflection factor determined at three different frequencies of 0.8, 1.0 and 1.2 MHz (i.e., $r(0.8)$, $r(1.0)$ and $r(1.2)$) for Mix 3 as a function of time are shown in Fig. 3. The temperature of the mortar mixture is also plotted on the same graph for reference. It can be seen that within the range of frequencies sampled, the mortar exhibits a similar response. At all three frequencies, the amplitude reflection factor initially decreases in magnitude, reaches a minimum and then starts increasing. The amplitude factor response at all three frequencies also exhibit similar distinctive features. The points in time which correspond to noticeable changes in the reflection amplitude response have been labeled A and B on the response determined at 0.8 MHz. The observed trend in the early reflected wave amplitude up to Point B suggests a two-phase process. There is a decrease in the wave amplitude factor up to Point B with an observed change in the rate associated with feature A in the amplitude response. Point B corresponds with the minimum value in the $r(f)$ response. It can be seen that while the $r(f)$ determined at the three frequencies exhibits a similar response, the distinctive features in the response appear to occur earlier in time at lower frequencies.

A comparison of the amplitude reflection factor determined at 1 MHz, $r(1)$, for Mix 1 (control), Mix 2 (mix containing accelerator) and Mix 3 (mix containing retarder) is shown in Fig. 4. The initial and final setting times for each mix determined using the ASTM C 403 procedure are also marked on the reflection factor curves for comparison. It can be seen that the basic trends in the response of the three mixtures are similar. The rate of decrease in the amplitude reflection factor, however, is different for the three mixes. Mix 2/Mix 3 shows the influence of the accelerator/retarder and is hence shifted to the left/right. Thus, the amplitude reflection factor detects changes in the rate of hydration introduced by admixtures with a high level of sensitivity. Further, it is interesting to note that the occurrence of features A and B in the $r(1)$ response approximately correspond in time with the initial and final setting times, respectively.

The rate of decrease of the reflection factor corresponds with the rate of increase of acoustic impedance of the mortar and hence the rate of change in its stiffness. Values of $r(f)$ close to 1 indicate a fluid-like state. Comparing the response of the three mortar mixtures in Fig. 4, it can be seen that the value of $r(1)$ for the Mix 3/Mix 2, which contains a retarder/accelerator, is approximately equal to 1.0 for a prolonged/shorter period of time compared with the control, suggesting that Mix 3/Mix 2 stays fluid (and hence workable) for a longer/shorter time after mixing. The trends observed in the reflection factor suggest an initial decrease up to Point B followed by an increase in the value of $r(f)$. It is known that the stiffness of the mortar increases continually with time after setting. Therefore, the two-stage response exhibited by $r(f)$, where an initial decrease is followed by a subsequent increase, requires careful consideration. An interpretation of

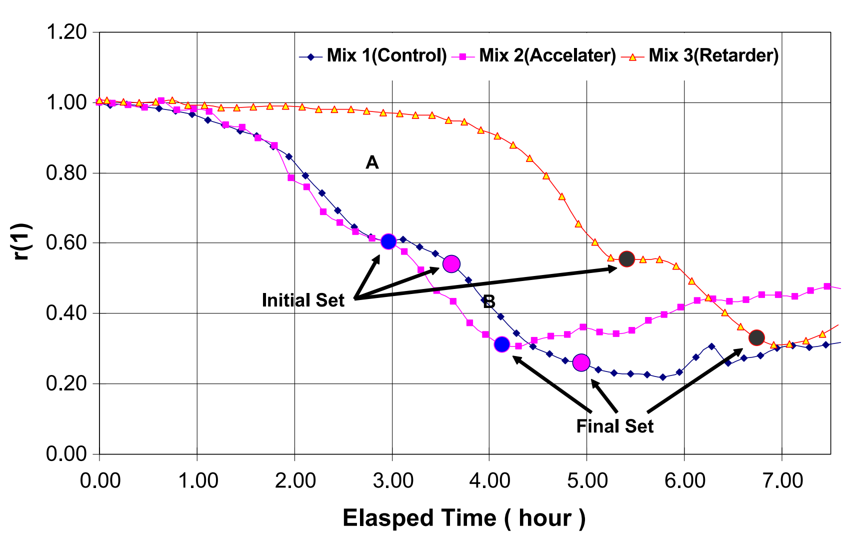

Fig. 4. Comparison of the amplitude reflection factor at 1 MHz, $r(1)$, for mortar mixtures containing accelerator (Mix 2) and retarder (Mix 3).

this observed trend in the $r(f)$ in terms of changes in material properties of mortar is attempted in the next section.

4.2. Interpretation of test results

Changes in the amplitude reflection factor are produced by changes in the acoustic impedance of the mortar with time. An interpretation of the observed trends in the amplitude reflection factor is now attempted through an analysis considering a simplistic representation of the mortar and the PMMA as visco-elastic materials. The shear modulus of a visco-elastic material is represented by a complex number, i.e., the shear modulus (G) has real and imaginary parts which are equal to the storage (G') and loss moduli (G''), respectively ($G=G'+iG''$). The storage and the loss moduli are also referred to as the elastic and the viscous component of the shear stiffness. The change in the amplitude of an incident wave produced by reflection of the shear waves at the interface between two visco-elastic materials given by the amplitude reflection factor, $r(f)$, is now obtained as

$$r(f)=\left|\frac{Z_2^*-Z_1^*}{Z_2^*+Z_1^*}\right| \tag{5}$$

where Z_2^* and Z_1^* are the acoustic impedances, respectively, of mortar and PMMA, which are complex numbers, and $r(f)$ is the magnitude of the complex number obtained by the expression in Eq. (5) [6]. The acoustic impedances, Z^*, of the mortar and the PMMA can be represented as shown below

$$Z_i^*=\sqrt{\rho_i(G_i'+iG_i'')}=Z_i'+iZ_i''=|Z_i^*|e^{i\phi_i/2} \text{ and }$$
$$\phi_i=\tan^{-1}\left(\frac{G_i''}{G_i'}\right) \tag{6}$$

where i equal to 2 and 1 applies respectively to mortar and PMMA, $|Z_i^*|$ is the magnitude of the acoustic impedance Z_i^* and ϕ_i is the shear phase angle of the material, which depends upon the ratio of the viscous and the elastic shear stiffness components. A larger value of the shear phase angle, ϕ_i, implies a larger magnitude of the viscous stiffness component relative to the elastic component and hence a more viscous response. The amplitude reflection factor can now be represented as

$$r(f)=\left|\frac{\left(|Z_2^*|/|Z_1^*|\right)e^{i\left(\frac{\phi_2-\phi_1}{2}\right)}-1}{\left(|Z_2^*|/|Z_1^*|\right)e^{i\left(\frac{\phi_2-\phi_1}{2}\right)}+1}\right|. \tag{7}$$

The values of $r(f)$ obtained using Eq. (7), as a function of $|Z_2^*|/|Z_1^*|$, are plotted in Fig. 5 for different values of $(\phi_2-\phi_1)$. The negative/positive values of $\phi_2-\phi_1$ correspond to the case when the shear phase angle of mortar is smaller/larger in magnitude when compared with that of PMMA. The $\phi_2-\phi_1=0$ line corresponds to the reflection the interface of two materials with identical ratios of the visco-elastic stiffness components. If ϕ_1 is considered equal to 0 (PMMA would then be an elastic material), then the $\phi_2-\phi_1=0$ curve represents the reflection at the interface of two elastic materials.

The theoretical trend shows that for any value of $\phi_2-\phi_1$, an initial decrease is followed by an increase in the value of $r(f)$ on increasing $|Z_2^*|/|Z_1^*|$. For all values of $\phi_2-\phi_1$, $r(f)$ attains its lowest value when $|Z_2^*|/|Z_1^*|$ is equal to 1. Therefore, for a fixed value of $\phi_2-\phi_1$, the amplitude reflection factor, $r(f)$, attains its lowest value, when the magnitude of the acoustic impedance of medium 2 is exactly equal to that of medium 1. For $\phi_2-\phi_1=0$, the amplitude of the reflected wave is equal to 0 when the acoustic shear impedance of medium 2 is equal to that of medium 1. In this case, no reflection is generated at the interface. For non-zero values of $\phi_2-\phi_1$, the minimum value of $r(f)$ is not equal to zero. This implies that if the shear phase angles of the materials forming the interface are not equal, on increasing $|Z_2^*|/|Z_1^*|$, the reflected wave would decrease, reach a non-zero minimum and then start increasing in amplitude.

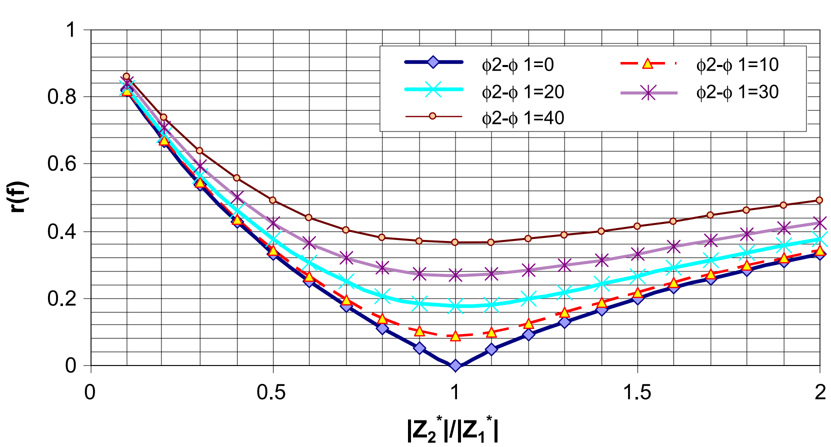

Fig. 5. The variation in $r(f)$ as a function of ratio of the magnitudes of the acoustic impedances of the two media for $(\phi_2-\phi_1)$ equal to 0°, 10°, 20°, 30° and 40°.

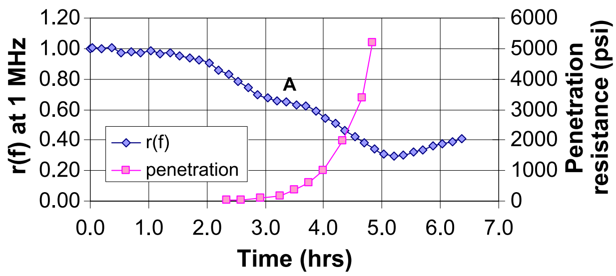

Fig. 6. A comparison between the measured $r(f)$ response at 1 MHz and the pin penetration resistance as a function of time.

The experimentally observed changes in $r(f)$ can now be interpreted using the results of the theoretical analysis and available results in the literature. After mixing, initially, the magnitude of acoustic impedance of mortar is considerably smaller than that of PMMA. With time, the mortar mixture exhibits an increase in stiffness. The stiffening of the mortar results in an increase in the magnitude of the acoustic impedance of mortar, $|Z^*_{\mathrm{mortar}}|$, relative to that of PMMA, $|Z^*_{\mathrm{PMMA}}|$. The analysis suggests that $r(f)$ will initially decrease, reach a minimum and start increasing in magnitude. The exact change in $r(f)$ for a given mortar mixture would depend upon the change in its shear phase angle with time. This change in the shear phase angle is brought about by a relative change in the viscous and the elastic components of the shear modulus of the material. Using low-frequency oscillatory shear measurements of cement pastes, it has previously been shown that with hydration, there is a continuous decrease in the measured phase angle of the material [12]. From the results of the analytical analysis shown in Fig. 5, since on decreasing the phase angle, the values of $r(f)$ are lower, it can be deduced that the minimum value of $r(f)$ will occur when $|Z^*_{\mathrm{mortar}}|/|Z^*_{\mathrm{PMMA}}|$ is approximately equal to or greater than 1. Thus, for a given mortar mixture composition, the minimum value of $r(f)$ provides a convenient point of reference when its acoustic impedance is approximately equal to that of PMMA. Since the acoustic impedance is a product of shear modulus and the density of the material, it can be inferred that for mortar of a given composition, the absolute value of the shear modulus of the mortar is a fixed percentage of that of PMMA at Point B of the measured reflection factor response.

4.3. Discussion

Feature A in the $r(f)$ response indicates a period of time when there is a change in the rate of decrease of $r(f)$. The observed trend in the decrease of $r(f)$ indicates a change in the mechanism that produces an increase in the stiffness of mortar. A comparison between the observed decrease in $r(1)$ (r(f) determined at 1 MHz) in the first few hours and the increase in penetration resistance of the same mix determined using the ASTM C 403 procedure is shown in Fig. 6. Comparing the response of $r(1)$ with the increase in penetration resistance it can be seen that feature A in the $r(1)$ response occurs approximately at the time when penetration resistance starts to increase. This suggests the possibility of using this feature in the $r(1)$ response to approximately identify the initial setting time. From the numerical derivative of the $r(1)$ response, an inflection point was consistently observed to occur in the region associated with feature A. The time of occurrence of the inflection point in the $r(1)$ response was identified using a numerical derivative procedure. The inflection point associated with feature A determined using the numerical procedure from the response at 1 MHz was found to consistently provide good agreement with the initial setting time. A comparison between feature A in amplitude reflection factor response and the initial setting time are shown in Table 2. A very close agreement between the two values can be observed. Thus, the inflection point associated with feature A in the $r(1)$ response provides a convenient method to determine the initial setting time of concrete. An interesting point of observation which results from this study is that while the setting behavior assessed using mechanical penetration tests exhibits a continuous increase, the behavior assessed using low-stress amplitude, high-frequency (1 MHz) waves exhibits a two-stage response through setting.

From the previous analysis, it has been established that Point B in the $r(f)$ response occurs when the acoustic impedance of mortar is approximately equal to that of PMMA. Thus, Point B represents the time when the mortar mixture attains sufficient stiffness in comparison with PMMA. A comparison between the final setting time determined using ASTM C 403 and Point B in the amplitude response at 1 MHz is shown in Table 2. It can be seen that there is a good correspondence between the time for occurrence of Point B and the final setting time. Furthermore, Point B in the wave response curve consistently occurs later in time than the time of final setting obtained using the ASTM C 403 procedure.

Table 2
Comparison between the time for occurrence of features A and B in the $r(1)$ response and the initial and final setting times

Mix number	Name	$r(1)$ response		Setting time (ASTM C 403)	
		Point A (h)	Point B (h)	Initial set (h)	Final set (h)
1	Normal	3.11	5.22	3.67	4.83
2	Accelerator	2.97	4.13	3.08	4.17
3	Retard	5.41	6.91	5.17	6.65
4	AE	3.29	4.62	3.33	4.33
5	Fly ash	4.73	6.56	4.20	5.72
6	SF	4.48	6.48	4.42	5.83
7	WC_35	2.63	4.80	2.50	3.75
8	WC_45	3.55	5.38	3.88	5.08
9	WC_50	3.71	5.55	4.08	5.33

Thus, the acoustic impedance of the mortar is approximately equal to that of PMMA after the time of final setting.

In the previous analysis, it has been shown that the observed changes in the amplitude reflection factor, $r(f)$, correspond to changes in the stiffness of the mortar relative to that of PMMA. From the experimental response shown in Figs 3 and 4, it can be seen that there is a significant decrease in the measured amplitude reflection factor up to the time of initial setting. Since the initial setting time approximately corresponds with the emergence of a solid phase, the changes in $r(f)$ up to the occurrence of feature A in the $r(1)$ response correspond to changes in the stiffness in the fluid stage. The response of $r(f)$ between Points A and B in $r(1)$ response provides an indication of stiffness changes through setting. For a mortar of a given material composition, Point B in the $r(f)$ response occurs when the shear stiffness of the mortar is a fixed percentage of that of PMMA. Further changes in $r(f)$ after B correspond to strength gains observed in the early ages after setting.

5. Conclusions

An ultrasonic technique for continuous monitoring of cementitious materials after mixing, through setting and early strength gain is reported in this paper. In the method described above, changes in stiffness of mortar reflected in its acoustic impedance are monitored relative to that of PMMA. The amplitude of the waves reflected at the PMMA–mortar interface, $r(f)$ is used to study the changes in the acoustic impedance of mortar. The ultrasonic measurements are shown to be sensitive to changes in the material properties from a very early age. Based on the results presented in this paper, the following conclusions can be drawn:

(a) There is a significant change in the acoustic impedance and hence the shear modulus of the mortar relative to PMMA before initial set. These changes correspond to the changes in the acoustic impedance of the mortar before set.

(b) Changes in the stiffness of mortar produce an initial decrease in the amplitude of the reflected waves at 1 MHz. The amplitude of the reflected waves reaches a minimum and then starts increasing as the mortar stiffens with age. At the minimum point in the $r(f)$ response, the acoustic impedance of mortar is approximately equal to or larger than that of PMMA. For a mortar of a given material composition, the minimum point in the $r(f)$ response occurs when the stiffness of the mortar is a fixed percentage of that of PMMA. The minimum point in the $r(f)$ response consistently occurs after the final setting obtained using the ASTM C 403 procedure.

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