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Application of Ultrasonic Wave Reflection for Setting and Stiffening of Cement Paste

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ABSTRACT

The objective of this research is to use ultrasonic wave reflection (UWR) measurement for monitoring the stiffening and setting process of portland cement paste. The research focuses on the very early hydration period, especially during the first hours after mixing. For such purpose, it is necessary to obtain higher measurement sensitivity, in this case by employing a buffer (high impact resistance polystyrene or HIPS) with acoustic impedance that is close to that of cement paste. The use of the HIPS buffer together with a new signal compensating method provided sensitive, accurate and reliable measurements on the stiffening and setting process of early-age cement paste. The information from P-wave and S-wave was used to understand the effect caused by segregation and flocculation of the cement paste. Finally, the criteria for determining the initial and final set of cement paste were suggested.

1. INTRODUCTION

Generally, two tests have been widely used to determine setting time of cement paste: the Proctor test (ASTM C403) and the Vicat test (ASTM C 191). The Vicat test uses mixtures with dry consistency, and gives almost no information on gradual changes due to the hydration. For this reason the Proctor test (C 403) was used for monitoring stiffening of cement paste in our previous work [Chung 2004; Struble et al. 2001]. The ASTM Proctor test (C 403) was able to measure stiffening until final set, but this technique measures data at discrete time periods, and it is likely to miss hydration events in between each measurement that may be important to understand stiffening process of cement paste. The dynamic rheology technique was also utilized to correlate hydration and rheological behavior of cement paste [Zhang 2001; Lei 1995], but some cement pastes quickly reached the stress limit of the instrument [Chung 2004], making

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it impossible to obtain meaningful data for understanding the stiffening progress of cement paste. Thus, the ultrasonic wave reflection technique (UWR) was chosen as an alternative.

UWR measures reflected ultrasonic wave energy at the interface between a buffer material and cement paste. As the cement paste hydrates and its acoustic impedance increases to be closer to that of buffer material, more ultrasonic wave energy passes though the cement paste and less reflects back to the transducer, allowing continuous measurements on microstructural changes due to hydration. The main objective of this research is to develop and extend the use of UWR to monitor stiffening process of cement paste during the very early hydration period. The sensitivity of the UWR measurements depends on the choice of buffer, so a low acoustic impedance buffer, high impact resistant polystyrene (HIPS), was selected.

2. EXPERIMENTAL PROCEDURES

2.1. Preparation

Cement used in this study was a Type I portland cement, conforming to ASTM C 150 Standard Specification for Portland Cement. Cement pastes were prepared by mixing 1000 g cement and 500 g water making w/c of 0.5. The mixing procedure, generally following that in ASTM C 305, was to mix paste for 30 sec at the lowest speed level (140±5 rpm), to stop the mixer for 30 sec to 60 sec and scrape down cement paste adhering to the side of the mixing bowl, then to mix for 90 sec at the lowest speed level. Throughout sample preparation and testing, the temperature of the laboratory was maintained at 20~22ºC. Mixing water was stored in the laboratory for a day to make the temperature of water equilibrated to that of laboratory.

2.2. Measurement

Cement paste was poured into the container made of HIPS buffer, the reflection coefficient was measured using contact type 2.25 MHz P-wave and S-wave transducers attached to the bottom of the container. A HIPS buffer was selected because it has a relatively low acoustic impedance (2.27 MRayls), thus providing sensitivity to small changes in solution acoustic impedance. Other materials were also used to compare the effect of buffer type on UWR measurements. The thickness of the buffers were 6.25-mm for HIPS, 6.25-mm for PMMA, 12.5-mm for aluminosilicate, 18.6-mm for glass, and 19.6-mm for 304 stainless steel, respectively. The appropriate thickness of a given buffer depends on two issues: 1) minimizing the travel distance of ultrasonic wave pulse (to minimize the energy loss during travel) and 2) capturing proper time domain signals without overlapping or clipping of the individual reflected wave pulses.

Solid phenol salicylate couplant was used to mount the transducer to the buffer. The transducers were connected to two Panametrics 5077 pulser/receiver units. The pulser/receiver unit is connected to a computer equipped with a NI 5102 PCI type digitizer (8–bit, 2–channel), using a sampling rate of 10 MHz for each transducer. The computer program LabView was used to control, collect, and process data. Once the data are obtained, the wave reflection coefficient is calculated using single pulse compensating procedure, described in our earlier work [Chung et al. 2010].

3. RESULTS AND DISCUSSION

Figure 1 shows the ultrasonic S-wave reflection coefficient values of w/c 0.5 cement paste with various buffers. It was observed that the use of different buffers changes the nature of the UWR curve. HIPS and PMMA showed an inversion. However, aluminosilicate (AS), glass, and 304 stainless steel (SS) did not show the inversion. When HIPS buffer showed an inversion at about 600 minutes, 304 SS showed reflection coefficient value of 0.96. This huge difference came from the differences in the S-wave acoustic impedances of the buffers, which are 0.998 MRayls for HIPS and 25.295 MRayls for 304 SS, respectively [Chung et al. 2012]. It should be noted that HIPS buffer has the lowest acoustic impedance (ZB) among tested buffers, and thus is the most sensitive to small changes associated with the hydration of cement paste.

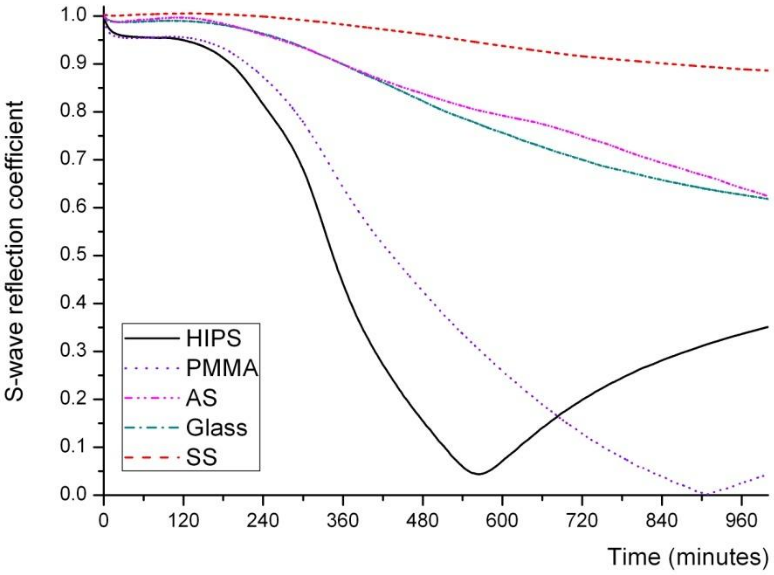

Figure 1. Ultrasonic S-wave reflection curves with various buffer types [replotted from Chung et al. 2012].

To observe the test reproducibility with HIPS buffer, the same mixture was tested on seven separate occasions, and the results are presented in Figure 2. As shown from Figure 2, the seven replicate w/c 0.5 cement paste specimens generally followed a similar trend although some scatter was seen in the data. An initial decrease was consistently observed during the first 20 minutes. It was caused by reforming particle to particle contact (flocculation of cement paste) and some amount of segregation in cement particles as discussed in our earlier publication [Chung et al. 2011]. The S-wave response then shows a slight decrease or a plateau for 100 minutes, and starts to show decrease substantially at about 120 minutes. The rapid drop in S-wave reflection response was associated with the microstructural developments due to hydration. The rapid drop in S-wave reflection was associated with the onset of stiffening [68]. It was referred as the onset of stiffening because it was later than initial set measured by traditional penetration based measurements [Chung et al. 2012].

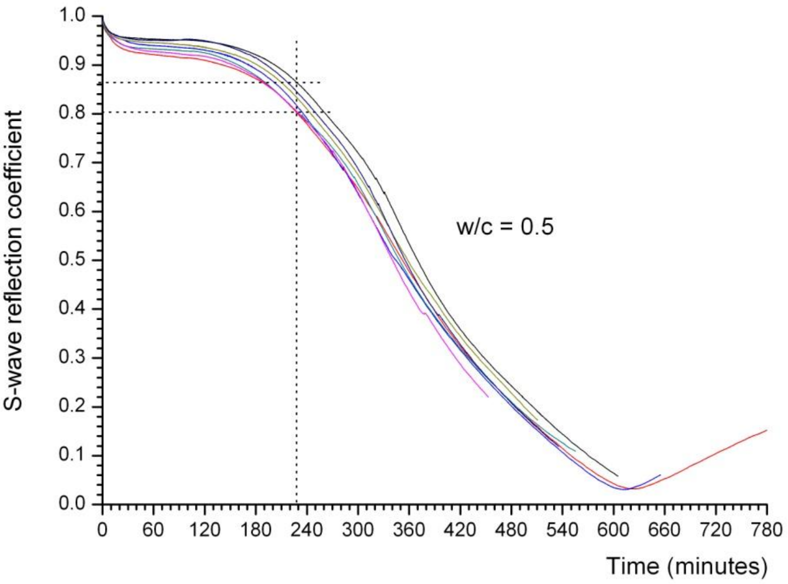

Figure 2. Repeated ultrasonic S-wave reflection responses for w/c 0.5 cement paste (replotted from Chung et al. 2012)

4. CONCLUSIONS

The changes in UWR with hydrating cement paste were associated with material stiffening and setting. The UWR responses from cement paste were determined at various w/c and with various buffer materials. Early changes, during first 20 minutes after mixing, were found to be associated with segregation (detected principally by P-wave UWR response) and flocculation (detected principally by S-wave UWR response) processes, respectively. Such observations are enabled by the choice of a low acoustic impedance buffer (HIPS), and it can be concluded that the use of the HIPS buffer improves the sensitivity of measurements on early age cement paste.

REFERENCES

Chung, C-.W., 2004, “Effect of Premature Stiffening and Its Relationship to Flow Behavior of Cement Paste”, MS thesis, Urbana (IL): University of Illinois at Urbana-Champaign.

Struble, L., J. Kim, T.–Y. and Zhang, H. (2001), “Setting of Cement and Concrete”, Cement, Concrete, and Aggregates, 23 (2), 88-93.

Zhang, H. (2001), “Using Dynamic Rheology to Explore the Microstructure and Stiffening of Cementitious Mixtures”. Ph.D. Dissertation, University of Illinois at Urbana Champaign.

Lei, W. G. (1995), “Rheological Studies and Percolation Modeling of Microstructure Development of Fresh Cement Paste”. Ph.D. Dissertation, University of Illinois at Urbana-Champaign.

Chung, C. W. Popovics, J. S. and Struble, L. J. (2010), “Using Ulltrasonic Wave Reflection to Measure Solution Properties”, Ultrasonics Sonochemistry, 17, 266-272.

Chung, C. W. Popovics, J. S. and Struble, L. J. (2011), “Flocculation and Sedimentation of Suspensions Using Ultrasonic Wave Reflection”, J. Acoust. Soc. America, 129 (5), 2944-2951.

Chung, C. W. Suraneni, P. Popovics, J. S., and Struble, L. J. (2012), “Setting Time Measurement Using Ultrasonic Wave Reflection”, ACI Mater. J. 109 (1), 108-117. Jan-Feb 2012.