Early-Age Properties of Cement-Based Materials. II: Influence of Water-to-Cement Ratio

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Abstract: The influence of water-to-cement mass ratio (w/c) on early-age properties of cement-based materials is investigated using a variety of experimental techniques. Properties that are critical to the early-age performance of these materials are tested, including heat release, semiadiabatic temperature, setting time, autogenous deformation, and strength development. Measurements of these properties using a single cement are presented for four different w/c, ranging from 0.325–0.425. Some of the measured properties are observed to vary widely within this range of w/c ratios. The heat release and setting time behaviors of cement pastes are contrasted. While early-age heat release is relatively independent of w/c, the measured setting times vary by several hours between the four w/c investigated in this study, indicating the fundamental differences between a physical process such as setting and heat release, which is purely a quantification of chemical reaction. While decreasing w/c certainly increases compressive strength at equivalent ages, it also significantly increases autogenous shrinkage and may increase semiadiabatic temperature rise, both of which can increase the propensity for early-age cracking in cement-based materials.

Introduction

One of the major variables influencing the performance of a concrete mixture is its water-to-cement mass ratio (w/c). From the earliest days of concrete, the major influence of w/c on compressive strength has been recognized (Feret 1897; Abrams 1918). Because the w/c directly controls the volume of water available for hydration per unit volume of cement and also establishes the initial interparticle spacing between cement particles, it will influence a wide variety of early-age properties such as setting time, semiadiabatic temperature rise, and autogenous shrinkage. The latter two variables can potentially be major contributors to early-age cracking of cement-based materials (ACI 2009). This paper contrasts these performance properties for four different w/c cement pastes and mortars prepared from a single cement. In Part I of this series, a similar study was conducted with cement fineness as the independent variable (Bentz et al. 2008).

Experimental Setup

For this study, a single Type I/II portland cement was used while the w/c of the cement pastes and mortars was set at values of

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0.325, 0.350, 0.400, and 0.425. This range of w/c was selected based on having mixtures that did not exhibit excessive bleeding while still providing adequate workability without the addition of any chemical admixtures. The measured oxide composition and Bogue potential phase composition, as provided by the manufacturer, are listed in Table 1. A Blaine fineness of 380 m^2/kg was reported by the manufacturer. The cement specific gravity was measured at the National Institute of Standards and Technology using ASTM C188 (ASTM International 2003); two replicates each gave a value of 3,220 kg/m^3. The measured particle size distribution (via laser diffraction) of the selected cement is given in Fig. 1.

For measurements of calorimetry and setting time, cement pastes were prepared using a high shear blender. Mortars were prepared according to the ASTM C 305–06 procedures (ASTM International 2006a) using a blend of four sands, each with a specific gravity of 2,610 kg/m^3. The mortar mixture proportions for the four different w/c mixtures are provided in Table 2. In each mortar mixture, the same volume fraction of sand (about 54.8%) was maintained; the water absorptions of the silica sands were assumed to be negligible. The $w/c=0.325$ mortar mixture exhibited a very low workability that made it somewhat difficult to prepare the mortar cubes and corrugated tubes for strength and autogenous deformation evaluations, respectively. The average measured air contents of the fresh mortars was 3.1%, with a range of 2.7–3.4% observed for the four different w/c mixtures.

The following measurements were conducted on the cement paste and mortar specimens:

1. Isothermal calorimetry—the heat of hydration was measured during the course of 7 days on premixed (as opposed to being mixed in situ in the calorimeter cells) sealed cement paste samples with a mass between 4.9 and 5.4 g or about 8 g of premixed sealed mortars using a TAM (TA Instruments, New Castle, Del.) air calorimeter. (Certain commercial products are identified in this paper to specify the materials used and procedures employed. In no case does such identification

Table 1. Cement Oxide Composition and Bogue Potential Phase Mass Fractions as Provided by the Cement Manufacturer

Oxide or property	Mass fraction
CaO	0.6301
SiO_2	0.2027
Al_2O_3	0.0473
Fe_2O_3	0.0351
SO_3	0.0286
MgO	0.0315
Na equivalent	0.54
Bogue—C_4AF	0.107
Bogue—C_3A	0.066
Bogue—C_3S	0.575
Bogue—C_2S	0.148

Fig. 1. Measured differential particle size distribution for the cement. The *x*-axis indicates an equivalent spherical particle diameter and the *y*-axis the mass percent at each given diameter. The shown results are the average of six individual measurements and the error bars (one standard deviation) would fall within the size of the shown symbols.

imply endorsement by the National Institute of Standards and Technology nor does it indicate that the products are necessarily the best available for the purpose.) To provide an indication of variability, two specimens from the same batch were evaluated in neighboring calorimeter cells for each experiment. In the results to follow, results are normalized per unit mass of cement in the sample.
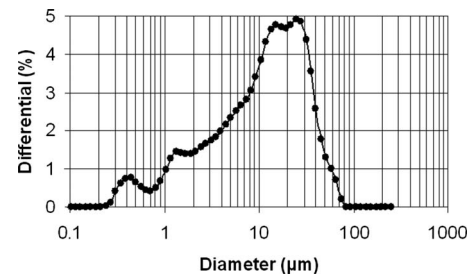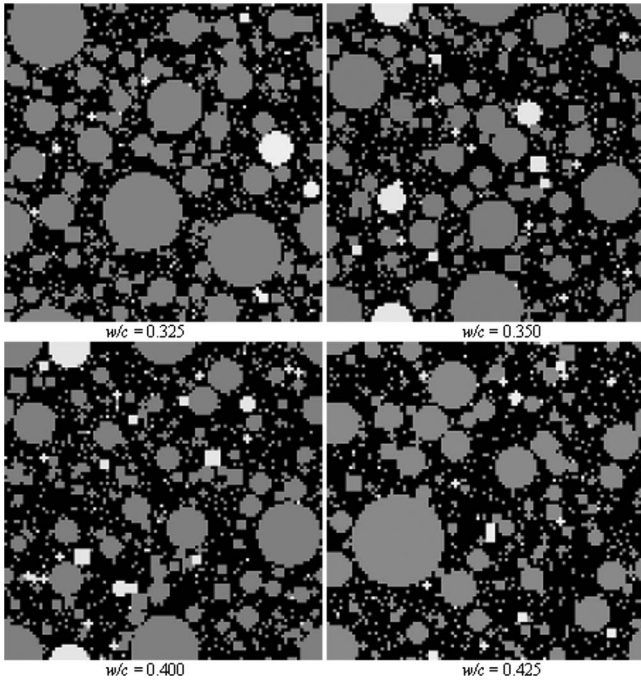
2. Semiadiabatic calorimetry—the semiadiabatic temperature was measured during the course of 3 days on single sealed cement paste specimens with a mass of approximately 330 g using a custom-built semiadiabatic calorimeter unit (Bentz and Turpin 2007). Replicate specimens from separate batches have indicated a standard deviation of $1.4°C$ in the maximum specimen temperature achieved during a 3-day test.

3. Time of setting—measured on single sealed cement paste specimens based on penetration of the Vicat needle according to ASTM C 191 (ASTM International 2007a); in the ASTM C 191 standard, the single laboratory precisions are listed as 12 and 20 min for initial and final time of setting, respectively.

4. Compressive strength—measured at 1, 3, 7, and 28 days on mortar cube specimens cured in a saturated calcium hydroxide solution, according to the procedures in ASTM C 109 (ASTM International 2007b) but with a loading rate of 20.7 MPa/min, switching to deformation control (at the instantaneous deformation rate) once a stress of 13.8 MPa was reached. Three specimens prepared from a single batch were evaluated at each time, with the averages and standard deviations provided in the results to follow.

5. Autogenous deformation—measured on triplicate or duplicate sealed mortar specimens prepared from a single batch sealed in corrugated tubes (Jensen and Hansen 1995) (proce-

dure is currently being standardized in ASTM subcommittee C09.68). In the draft standard, the single laboratory precision is listed as 30 microstrains for mortar specimens.

The initial three-dimensional microstructures based on these four different w/c will be significantly different. To illustrate this point, Fig. 2 shows two-dimensional cross section images from simulated three-dimensional starting microstructures (Bentz 1997) where the assumed spherical cement particle diameters follow the size distribution provided in Fig. 1 and the particles have been flocculated in three-dimensional space (since no chemical admixtures were used in the current study). Each two-dimensional image is 100 $\mu m \times 100$ μm. Qualitatively, the interparticle spacing (initial pore size) looks to be greater in the higher w/c microstructures. Using software available at http://ciks.cbt.nist.gov/cementspacing.html, the water-to-cement distance function (Bentz and Aitcin 2008) was simulated for each of these mixtures, with particle placement at totally random locations beginning with the largest particles. The water-to-cement distance function measures the volume fraction of the total water that is within a given distance of a cement particle surface. The results for this fractional volume of water are provided in Fig. 3. The distance values for volume fractions of water of 0.9 and greater provide an indication of the sizes of the initial pores that will be emptied during self-desiccation, for instance (Bentz and Aitcin 2008). Thus, this interparticle spacing (pore size) will have a large influence on the development of autogenous stresses and strains within the hydrating cement paste and mortar specimens (Bentz et al. 2001; Baroghel-Bouny et al. 2006).

Table 2. Mortar Mixture Proportions Used in the Study

Material	$w/c=0.325$ (g)	$w/c=0.350$ (g)	$w/c=0.400$ (g)	$w/c=0.425$ (g)
Type I/II cement	1,800.0	1,800.0	1,800.0	1,800.0
Water	585.0	630.0	720.0	765.0
F95 fine sand[a]	903.7	940.5	1,008.9	1,047.4
Graded sand (ASTM C 778)[b]	686.8	714.8	766.8	796.0
20–30 sand (ASTM C 778)[b]	686.8	714.8	766.8	796.0
GS16 coarse sand[a]	1,337.5	1,391.9	1,493.2	1,550.1

[a]F95 and GS16 correspond to sand supplier designations.
[b]ASTM International 2006b.

Fig. 2. Single two-dimensional cross-sectional images (100 μm × 100 μm) from three-dimensional simulated starting microstructures for cement pastes for the four different w/c investigated in this study (cement particles are gray and gypsum particles are white)

Results and Discussion

Compressive Strength

The compressive strength results are considered to provide a frame of reference in which to consider the other early-age property measurements. As shown in Table 3, the two lowest w/c consistently produce higher compressive strengths at all four testing ages. At the ages of 1 and 7 days, the strengths of the w/c =0.325 mortar cubes are slightly lower than those of the w/c =0.350 cubes. This may be due to the low workability of the w/c=0.325 mortar and difficulty in properly compacting the fresh mortar mixture into the cube molds or to a reduction in the achievable degree of hydration due to space limitations as w/c decreases below about 0.38 in a saturated system (Taylor 1997).
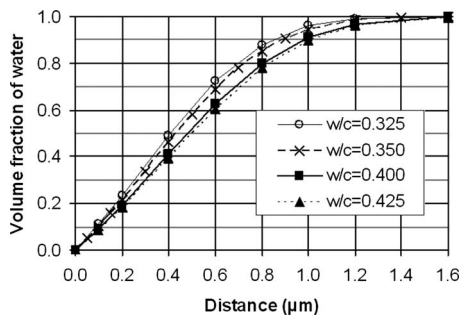
Fig. 3. Water-to-cement distance functions (Bentz and Aitcin 2008) computed for three-dimensional starting microstructures (200 μm × 200 μm × 200 μm) for the four different w/c cement pastes investigated in this study

Table 3. Compressive Strength Results for Mortar Cubes

| Age (d)	w/c=0.325 (MPa)	w/c=0.350 (MPa)	w/c=0.400 (MPa)	w/c=0.425 (MPa)
1	33.8 (1.0)[a]	36.2 (1.4)	25.2 (0.4)	20.9 (0.1)
3	56.1 (1.8)	55.6 (2.4)	47.6 (1.5)	41.0 (0.4)
7	64.0 (4.4)	64.8 (1.0)	55.1 (1.6)	50.8 (1.0)
28	81.8 (4.4)	78.5 (2.2)	72.8 (1.4)	68.7 (3.4)

[a]Numbers in parentheses indicate one standard deviation as determined for the three replicate specimens tested at each age.

### Isothermal Calorimetry and Setting Characteristics

Next, the heat release properties of the various mixtures are considered. The isothermal calorimetry results for the four different w/c pastes are provided in Figs. 4 and 5, in terms of the cumulative heat release and the heat release rate at early ages, respectively. For the cumulative heat release results, when normalized by the mass of cement in each sample to relate heat release to degree of cement hydration, the four curves overlap one another for the initial 12 h before approaching different values at later ages. It is well known that for w/c below about 0.44, there is insufficient water to provide complete hydration of the anhydrous cement under sealed curing conditions (Taylor 1997). Because the isothermal calorimetry specimens are sealed with no external curing water supplied, they will self-desiccate due to the chemical shrinkage that accompanies the cement hydration reactions (Bentz 1997). This will decrease the ongoing hydration rates, an effect that will be more pronounced in lower w/c pastes. Thus, in Fig. 4, the heat release curves for w/c=0.400 and w/c=0.425 basically
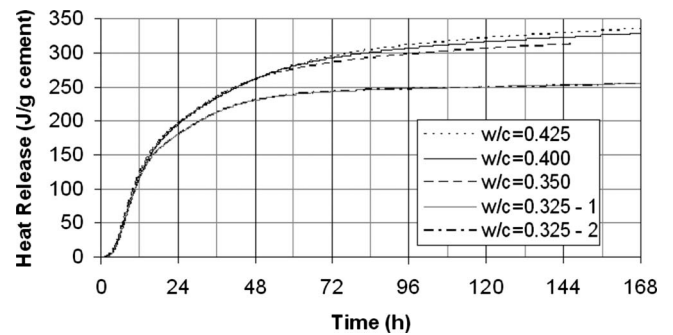
Fig. 4. Cumulative heat release for 7 d from isothermal calorimetry for the cement pastes with four different w/c. Two curves that basically overlap one another are shown for the w/c=0.325 paste to provide an indication of variability between duplicate specimens.
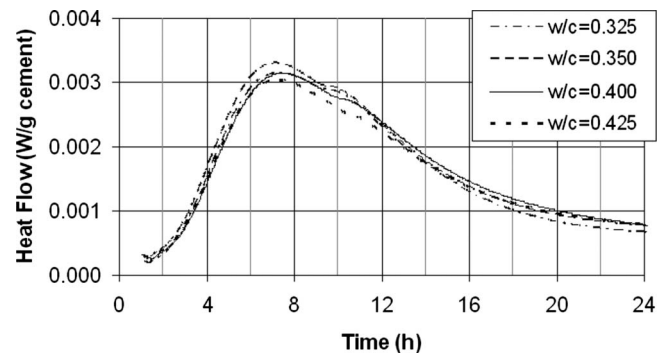
Fig. 5. Early-age heat release rates from isothermal calorimetry for the cement pastes with four different w/c
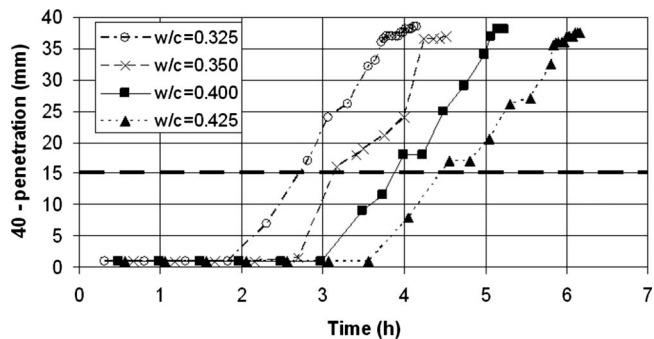
Fig. 6. Vicat setting (plotted as 40—needle penetration) results for the cement pastes with four different w/c; dashed horizontal line indicates initial set per the ASTM test method

Fig. 7. Early-age heat release rates from isothermal calorimetry for the mortars with four different w/c

overlap one another for the entire 7 days of hydration examined in this study, as during these first 7 days of hydration, neither paste becomes water limited. The $w/c=0.350$ paste breaks away from these two curves after about 2 days of hydration while the $w/c=0.325$ curve diverges from the others much earlier, after only about 12 h of hydration, due to an inadequate supply of water to maintain hydration.

The heat release rate curves for the first 24 h of hydration shown in Fig. 5 are quite similar for the four different w/c pastes examined in this study. Previously, Sandberg and Roberts (2005) have also observed quite similar heat release rate curves for pastes prepared with a single cement with w/c of 0.35, 0.40, and 0.45. While the heat release results are nearly independent of w/c for the pastes prepared in this study, the setting times of the pastes as assessed using Vicat needle penetration are strongly influenced by w/c, as shown in Fig. 6. The initial setting times (heavy dashed line in Fig. 6 indicating a penetration of 25 mm) are seen to vary by over 1.5 h for the range of w/c investigated in this study while the final setting times (no penetration) vary by about 2 h. Clearly, the degree of hydration necessary to produce setting (a physical process) is a function of w/c, with higher w/c requiring more hydration, likely due to their increased interparticle spacing (Figs. 2 and 3).

While it appears clearly from the results in Fig. 5 that isothermal calorimetry (a measure of chemical reaction) cannot provide a reliable indication of setting (a physical process), as shown in Fig. 6, for cement **pastes** of different w/c, some other research groups are currently using it as a technique to predict the setting times of mortars and concretes as measured using ASTM C403 (ASTM International 2006c). To investigate this apparent contradiction further, the measurements were repeated for mortar mixtures (as provided in Table 2) for the same values of w/c, using the ASTM C807 standard test method for time of setting of hydraulic cement mortar by modified Vicat needle (ASTM International 2005). The heat release rate results for these four mortars are provided in Fig. 7. For the mortars, as compared to the pastes in Fig. 5, a greater dispersion between the four curves is observed. Taking the first maximum in the derivative of the heat release rate curve as an indication of set, Fig. 8 provides a plot of these "thermal times" versus the times of set measured using the modified needle. The results are certainly more promising than in the case of cement pastes but do not fall on a 45° line, indicating that while correlation exists between the two times, they are not identical and cannot simply be used interchangeably. Results from other groups have indicated a similar correlation between ASTM C403 setting times and these thermal indicators of setting but
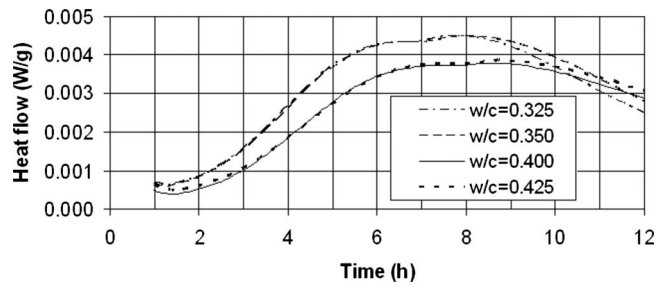
with relationships that differ significantly from one cement to another, further supporting the observation that the relationship between setting and thermal indications of hydration is not a simple identity function. In summary, based on the results presented in this study, for cement pastes prepared in the laboratory, isothermal calorimetry is not a reliable indication of setting time for w/c in the range of 0.325–0.425, as only minimal variation between the isothermal calorimetry curves is observed for w/c in this range. Conversely, for **mortars**, an adequate correlation is observed between setting time and the time of the first maximum in the derivative of the heat release rate but it is likely that calibration would be required to determine the specific correct relationship for each individual cement and possibly also for each cement-admixture combination. Recently, similar conclusions have been reached for cement pastes regarding chemical shrinkage measurements (another measure of chemical reaction) and setting time (Sant et al. 2008).

Semiadiabatic Temperature

The influences of w/c on thermal properties at early ages were further investigated using semiadiabatic calorimetry measurements on cement pastes. Semiadiabatic calorimetry may provide an indication of the susceptibility of a mixture to early-age thermal cracking (Bentz et al. 2008; ACI 2009; Bentz and Peltz 2008). The plots of temperature versus time for the four cement pastes are provided in Fig. 9. A change in w/c has several distinct influences on the semiadiabatic temperature curves. First, because water has a much larger heat capacity than anhydrous cement, 4.18 J/(g·K) versus 0.75 J/(g·K) (Bentz 2007), a smaller temperature rise would be expected when w/c is increased (Bentz
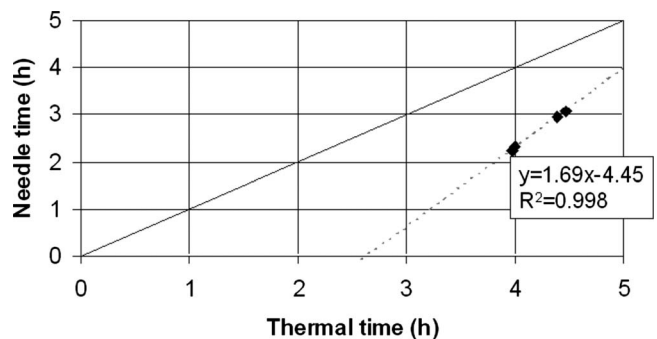
Fig. 8. ASTM C 807 time of set versus time of maximum in derivative of heat release rate for the four cement mortars with different w/c. Dark line indicates an identity relationship and dashed line indicates best linear fit.
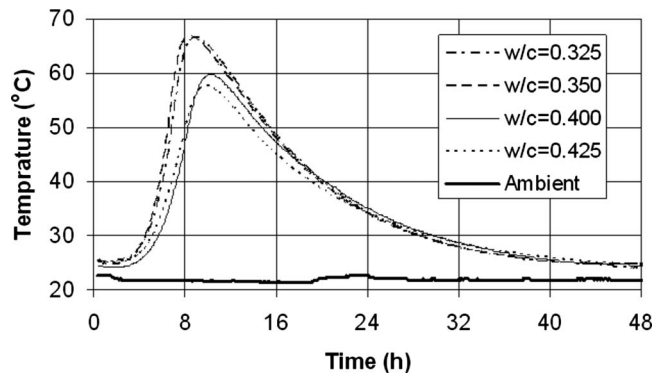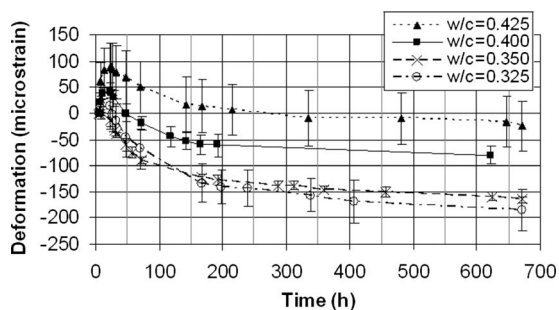
Fig. 9. Semiadiabatic temperature during the first 48 h of hydration for the cement pastes with four different w/c

Table 4. Net Autogenous Shrinkage Strains at 7 d for the Four Mortar Mixtures

Mortar mixture	$(\varepsilon_{min} - \varepsilon_{max})$ (microstrains)
$w/c = 0.325$	-151
$w/c = 0.350$	-127
$w/c = 0.400$	-100
$w/c = 0.425$	-75

and Peltz 2008). Second, less cement per unit volume also means less heat generation within the fixed volume semiadiabatic calorimeter specimen holder when w/c is increased. Conversely, the influences of w/c on the achievable long term degree of hydration as discussed above may result in less heat being produced once w/c falls below about 0.36. These effects are well illustrated by the results in Fig. 9 where peak temperature first increases as the w/c is lowered from 0.425 to 0.400 to 0.350 but then exhibits no further increase as w/c is further lowered to 0.325, as the lower heat capacity and higher cement content of the paste are finally offset by the decreased (achievable) hydration at the lowest w/c examined in this study.

Autogenous Deformation

Finally, the autogenous deformations of the four mortar mixtures are considered. Once again, as shown in Fig. 10, w/c has a large influence on this early-age property of cement-based materials. The autogenous deformation measured for each mortar mixture is a competition between an autogenous shrinkage due to self-desiccation in these sealed corrugated tube specimens and an expansive process that is not well understood but is conjectured to be due to ettringite and/or calcium hydroxide crystal formation and growth or perhaps imbibition of water by the cement hydration (gel) products (Bentz et al. 2001; Baroghel-Bouny et al. 2006). Expansion at early ages could also be due to thermal expansion as energy is released by the cement hydration reactions but for the 25-mm diameter corrugated tubes employed in this study, temperature changes at early ages should be less than $2\,°C$ according to the draft ASTM standard. In actuality, by inserting a

single thermocouple into the center of the corrugated molds, the following temperature changes were measured between the time of set and 24 h for the $w/c = 0.325$, 0.35, 0.4, and 0.425 mortars: 0.9, 1.0, 0.9, and $1.0\,°C$. A $1\,°C$ temperature change should result in an expansion/contraction of about 10 to 20 microstrains, much less than the expansions indicated in Fig. 10.

It is clear that as w/c decreases, the initial interparticle spacing decreases (Figs. 2 and 3), which will increase the magnitude of the autogenous stresses induced by the menisci in the partially water-filled capillary pores. It is less clear if the expansive processes are influenced by w/c but the results in Fig. 10 do clearly indicate that any change in early-age expansion with w/c is far overwhelmed by the increase in autogenous shrinkage as w/c is decreased. For each cement (having a given degree of fineness), there will be a critical w/c below which the measured long term (28 days) autogenous deformation will exhibit a shrinkage and above which an expansion will be measured (Baroghel-Bouny et al. 2006; Bentz et al. 2008). For the cement employed in this study, this "threshold" w/c (Baroghel-Bouny et al. 2006) would be slightly above 0.425, as the $w/c = 0.425$ mortar exhibited only a small autogenous shrinkage of about -30 microstrains after 28 days hydration.

Recently, Cusson has advocated the utilization of the (net) difference between the maximum (peak) and minimum (achieved post peak) deformations achieved during the first 7 days of curing as an effective measure of the risk of cracking in a concrete structure, as a concrete typically reaches a zero-stress state sometime after the peak (expansion) is achieved but prior to the autogenous deformation returning to zero (or achieving its minimum value) (Cusson 2008). Beyond this zero-stress time, tensile stresses develop and build under restrained conditions and may ultimately lead to early-age cracking if the tensile strength of the concrete is exceeded at any time (ACI 2009; Cusson 2008). For the four mixtures examined in this study, these values of net autogenous shrinkage, $(\varepsilon_{min} - \varepsilon_{max})$, are provided in Table 4. The magnitudes of these net autogenous shrinkage values monotonically decrease with increasing w/c, supporting the generally accepted hypothesis that lower w/c mixtures will have an increased autogenous shrinkage and likely be more susceptible to the early-age cracking that such shrinkage may induce if movement is restrained.

Fig. 10. Autogenous deformation results for the four mortar mixtures. Error bars indicate one standard deviation as determined for the triplicate specimens of each mixture.

Conclusions

A comprehensive study of a variety of early-age properties of cement pastes and mortars with w/c ranging from 0.325–0.425 has been conducted including compressive strength, isothermal calorimetry, semiadiabatic temperature, setting time, and autogenous deformation. These studies have indicated that:

- For cement pastes prepared in the laboratory, isothermal calorimetry may not be a reliable indication of setting time for w/c in the range of 0.325–0.425, as only minimal variation be-

tween the isothermal calorimetry heat flow curves is observed for w/c in this range.

- For mortars prepared in the laboratory, correlation can be established between isothermal calorimetry and setting time measurements for w/c in the range of 0.325–0.425 but the best fit relationship is not a simple identity function (e.g., slope greater than one and nonzero intercept).
- Decreasing w/c will increase the peak temperature achieved during semiadiabatic hydration except for w/c less than about 0.38, where the reduced degree of achievable hydration will limit the heat release and consequent temperature rise.
- Decreasing w/c increases the autogenous shrinkage of mortars cured under sealed conditions, both in terms of the absolute deformation achieved after 28 days and the net shrinkage experienced during the first 7 days of hydration.

Acknowledgments

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