

Stress induced dissolution and time-dependent deformation of portland cement paste

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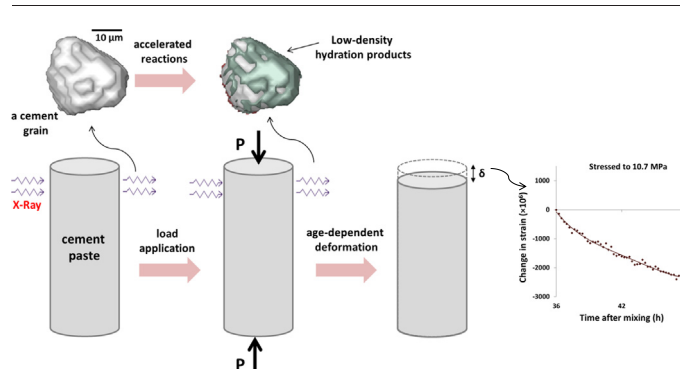
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HIGHLIGHTS

- In-situ observation made on cement paste suggested that the stress application changed the development of microstructure.
- The cement grains dissolved faster under stress.
- The accelerated reactions under load resulted in increase in stiffness and deformation of the samples.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 21 February 2018

Received in revised form 27 July 2018

Accepted 29 July 2018

Available online 31 July 2018

Keywords:

X-ray Computed Tomography

Microstructure evolution

Cement Hydration

Creep

Dissolution

ABSTRACT

While the stress-induced dissolution of various minerals has gained attention as an important time-dependent deformation mechanism, this has only sparingly been investigated in portland cement systems. In this paper, X-ray Computed Tomography (XCT) is used to make direct observations of the microstructural evolution in cement paste samples under different levels of stress during their first 60 h of hydration. Stiffness and creep measurements are also made while imaging the changes in the microstructure. The results show that stress applied at ages between 24 h and 60 h caused an increase in stiffness, increase in early age creep, and a statistically higher amount of dissolution of individual particles near the point of load application.

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1. Introduction

Recent publications suggest a significant coupling between the mechanics and the chemical composition during the early age reactions or hydration of portland cement paste [1–5]. If these properties can be coupled then this will provide a more accurate description of the hydration of portland cement paste and allow for improved control of this

important material. While stress induced dissolution has gained little attention as a mechanism for time-dependent deformation in cementitious materials, it has been studied extensively in various rocks and minerals [6–13]. Studies focused on gypsum pressured to 0.25–2.5 MPa [8, 12], halite stressed to 0.13–0.63 MPa [14], quartz [15, 16], limestone and sandstone pressured to 8.9–16.3 MPa [11], calcite with in-situ pressure between 434 and 808 MPa [7], and soluble salts (potassium alum and sodium chlorate) with pressure between 1 and 27 MPa [17] all suggest that stress induced dissolution is an important mechanism for viscoelastic/viscoplastic (VE/VP) behavior. It is logical

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that this could be important in cementitious systems because of the similarities in chemical composition and structure of these materials.

Other publications suggest that stress may impact the thermodynamic equilibrium, hydration kinetics, and/or the VE/VP properties of concrete [5, 18–21]. Previous work measured the impact of stress on the hydration of portland cement and C_3S by thermogravimetric analysis (TGA), BET surface area, and mercury intrusion porosimetry based pore-size distribution [5]. The work found up to a 14% increase in the degree of hydration of C_3S after 48 h for a paste with w/s (water-to-solid ratio) of 0.50 which was loaded hydrostatically to 6.8 MPa after 18 h compared to a non-loaded sample. Other work found that C_3S samples hydrated at room temperature with w/s = 0.55 that were cured under 85 MPa hydrostatic pressure reached the maximum conductivity after ≈ 100 min while the samples that were loaded at 35 MPa showed that the maximum conductivity was reached at ≈ 200 min. [18]; this indicates that the matrix may form faster under a higher pressure [18]. However, the conductivity could be impacted by other factors such as changes in dissolution rate from the increase in stress that would, in turn, change the pore solution chemistry. Regardless of this, the increase in stress caused the point of maximum conductivity to occur earlier than the sample with the lower load. Both of these experiments suggest that there is an increase in the rate of hydration with an increase in stress as well. Others have shown that stressed cement paste beams tested in 3-point loading showed approximately 8% higher Young's modulus than the non-loaded samples after 56 days of hydration [2]. This suggests that early age loading can cause changes in the mechanical properties of hardened cement paste.

Computational hydration models have been proposed to predict the influence of applied stress on VE/VP mechanisms in concrete. A recent study suggests that the dissolution of cement particles at early ages controls the time-dependent changes of the apparent VE/VP Poisson's ratio [3, 4, 21, 22]. These studies would greatly benefit from additional experimental data sets to guide these models.

Although these referenced studies suggest the importance of stress state on the physical properties of cement based materials, these studies are based on either computational models or experimental techniques that make bulk measurements of samples loaded during hydration with no direct observation of changes in the microstructure. This work aims to use X-ray computed tomography (XCT) as it is non-destructive and can allow for simultaneous measurement of the load deformation and the changes in the cement paste. Previous research used XCT to observe structural changes and void evolution in cement paste during the hydration of cement [23–27]. Also, higher resolution CT has been used to observe the 3D structure of individual C_3S particles during hydration [28–30]. This technique is a powerful tool to non-destructively visualize the internal structure of materials with a spatial resolution of as small as a few nanometers (nano tomography) [30, 31] to several microns (micro tomography) [32–34]. Another useful output from the data is that the variations of the gray values in the built images originate from the electron density and bulk density of the imaged material [28, 35, 36]. The gray values of the images can be used for quantitative evaluation of XCT datasets by separating the regions into different constituents. This process is called segmentation and this is important to obtain quantitative measures from the data.

This work uses XCT to provide quantitative insights into how stress changes the dissolution rate, early age creep, and stiffness over the first 60 h of cement hydration. This approach is unique because it can be used to make all of these measurements simultaneously on a loaded sample without disturbing it. Since this experimental method does not disturb the sample during hydration, it allows for critical measurements to be made without raising questions about testing artifacts.

2. Research significance

The ultimate objective of this research is to improve a developed computational model that links evolving system chemistry and

mechanics of cementitious materials, and to implement the model through a computational method that predicts the fully coupled evolution of microstructure and.

VE/VP properties of the cement paste. Any stress induced changes to the material microstructure and resulting time-dependent deformation will be predicted by the model. More details of this model that combines finite element methods with microstructure development simulations could be found in other and future publications [3, 4, 21, 22]. The results presented in the current paper will be used to verify and quantify the interconnection between chemistry and mechanics through phase dissolution in cement particles by time-stepping computed tomography.

The results give important insight to understand the evolution of constitutive properties and the underlying deformation mechanisms; this will ultimately enable design of concrete with greater strength, toughness, durability, and sustainability.

In the development of new cementitious systems, the current state of practice involves primarily trial-and-error experimentation and empirical modeling approaches to determine their performance and the impact of new binder systems. The proposed approach of linking coupled modeling to the evolution of important constitutive properties will ultimately allow a paradigm shift in how cementitious materials are developed and optimized. Therefore, this work can help to accelerate the development of cementitious materials engineered and optimized for particular applications and conditions from fundamental principles. The model framework and methodology will be extensible to other classes of materials such as biological materials and other ceramic materials.

3. Method and experiment

3.1. Materials

NIST 168 portland cement (OPC) from the Cement and Concrete Reference Laboratory (Frederick, Maryland) was used to make the paste samples. The cement composition is presented in Table 1. The analysis was completed by X-ray fluorescence (XRF) and X-ray diffraction (XRD) for mineralogical composition and ASTM C204 for Blaine fineness. The XRD pattern of this powder can be found in another publication [37].

Particle size distribution (PSD) was determined by Automated Scanning Electron Microscope (ASEM) [38]. The result showed that 95% of OPC particles are smaller than 8 μm in diameter. The PSD graph and more information about the ASEM technique can be found in other publications [26, 38, 39].

3.2. Sample preparation

The samples were produced with a water to solid ratio (w/s) of 0.50 (3.000 g of OPC powder and 1.500 g of deionized water) which is a typical w/s for portland cement paste. Water was added to the dry powder, and then they were mixed by a stainless steel rod 10 times clockwise and 10 times counterclockwise in a glass vial. Next, the vial was shaken with a speed of 1000 rpm for 3 min on a vortex mixer produced by Stuart (Staffordshire, UK). This mixing procedure ensures that the paste is well-mixed and consistent in all samples produced.

The paste was placed in cylindrical polyethylene molds with a diameter of 1.19 mm to 1.58 mm and a height of 9 mm. The variable diameter was caused by differences in manufacturing. The mold was then gently tapped by a steel rod to compact the samples. A wet paper towel was subsequently placed on the mold for 24 h to keep the sample moist and provide a favorable condition for curing. All cylindrical samples were carefully demolded after 24 h and covered by petroleum jelly to minimize the loss of moisture during data collection. This means that moisture can allow continued hydration. The entire diameter of the samples is imaged, but the analysis is performed on a region of interest that is 800 μm in diameter. This region was chosen to be far away from the boundaries, which may potentially have artifacts or inadvertently

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