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Early Age Autogenous Deformations of Cement-Based Materials

Multi-scale Micromechanics Modelling

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ABSTRACT: Autogenous shrinkage is considered to be one of the main phenomena involved in the early-age cracking of cementitious systems with low water-to-cement ratio. It results from the volume difference between the hydration products (hydrates) and the corresponding reactants (cement and water) and the self-desiccation of the porous network caused by the water consumption in the course of hydration. The objective of this paper is to model the autogenous shrinkage of a cement paste from the first contact between cement and water up to 2 days of hydration by means of a multi-scale homogenization model. The inputs of the model are the chemical composition and the Blaine fineness of cement, the mechanical properties of the main cement phases and the water-to-cement ratio (W/C). The outputs are the evolution of the cement phase volume fractions and chemical hydration reactions, the Young's modulus, the capillary tension, the chemical and the autogenous shrinkage. Numerical and experimental results are analysed in order to validate the model and propose improvements.

KEY WORDS: cement paste, autogenous shrinkage, early stage, modelling, experiments.

1. Introduction

Cement-based materials experience, at an early stage, dimensional variations caused by cement hydration heat release, physico-chemical evolution of the material and moisture transfers with the surrounding environment. In sealed conditions and without significant thermal variations, the free deformation observed is defined as autogenous shrinkage: it is caused firstly by the volume difference between the reaction products and the corresponding reactants (cement and water). This chemical shrinkage creates partially-saturated pores, within the material structure, which self-desiccate in the course of the hydration process. The self-desiccation of the porous network induces internal capillary tension and an external contraction of the material (Justnes *et al.*, 1996). Typical autogenous shrinkage curves usually present two main phases, separated by the setting period: an accelerated phase, corresponding to chemical shrinkage, also called Le Chatelier's contraction, which is followed by a second phase characterized by a flattening of the strain curve (self-desiccation shrinkage).

Models based on empirical relations (Tazawa *et al.*, 1995) have been developed with the aim of calculating the strains due to the autogenous shrinkage in cement pastes and mortars. However, these macroscopic models do not take into account the microstructure heterogeneities. The homogenization methods have been developed for the determination of the equivalent mechanical properties of heterogeneous materials (Bornert *et al.*, 2001). Recently, these methods have been applied to the calculation of the mechanical properties (Bernard *et al.*, 2003; Sanahuja *et al.*, 2007) and the autogenous shrinkage of cement pastes at an early stages (Pichler *et al.*, 2007). However, the model developed by Pichler *et al.* does not consider the chemical phase of autogenous shrinkage observed before setting.

In this work, we propose a multi-scales model to follow the evolution of cement paste autogenous strains from the end of mixing up to 48 h of hydration. From the mass fractions and mechanical properties of each main cement phase (C_3S , C_2S , C_3A , C_4AF and gypsum), the cement Blaine fineness and the water-to-cement ratio of the mixture, this model enables to calculate the chemical shrinkage and the self-desiccation shrinkage of cement paste. The paper firstly presents the experimental part of the work. The multiscales micromechanics model is then described. Finally, the numerical and experimental results are compared.

2. Experimental study

The experimental validation tests have been carried out on a cement paste with a W/C ratio of 0.40. Ordinary Portland cement (CEM I 42.5 from Saint Pierre La Cour plant) was used. Its Blaine fineness is 339 m²/kg and its Bogue's composition is 62% C_3S , 11% C_2S , 8% C_3A and 8% C_4AF . All testing – Vicat setting times,

chemical shrinkage, hydration degree and autogenous shrinkage – have been performed at a constant temperature of $20 \pm 0.5^\circ\text{C}$.

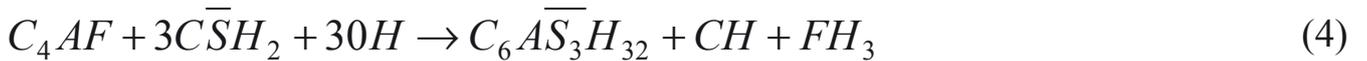
Vicat needle tests have been made in accordance with European standards NF EN 196-3. Chemical shrinkage was measured by hydrostatic weighing, ensuring permanent water saturation of the pore volume of the material, according to the protocol described by Mounanga *et al.* (2004). The hydration degree is obtained by loss on ignition method (Bouasker *et al.*, 2005): it consists of measuring the nonevaporable (chemically-bound) water content of the sample heated between 105 and 950°C . The hydration degree at time t is calculated as the ratio between the amount of chemically-bound water content at time t and the amount of water necessary for the complete hydration of cement.

Concerning autogenous shrinkage measurement, the hydrostatic weighing method has been used. An original testing device equipped with a rotary system has been developed in order to prevent the specimen from bleeding and sedimentation inside the latex membrane. Paraffin oil is used as the immersion liquid in order to limit liquid absorption by the sample.

3. Micromechanics modelling

3.1. Evolution of the phases during hydration

The modeling approach requires the determination of the evolutions of the volume and mass fractions of each phase of the material (unhydrated cement, water and hydrates) at an early stage. They are calculated on the basis of the chemical equations defined below:



Each cement component has a different hydration rate, which depends on its normalized affinity $\tilde{A}(\xi_X)$ and its characteristic time τ_X , according to Atkins (Atkins, 1994):

$$\tau_X \frac{d\xi_X}{dt} = \tilde{A}(\xi) \quad (9)$$

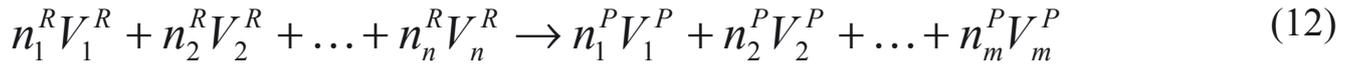
From the knowledge of the hydration degree (ξ_X), it is then possible to calculate the volumes of clinker phases (V_X) remaining in the mix (Bernard *et al.*, 2003):

$$V_X(t) = V_{C0} f_X (1 - \xi_X(t)) \quad (10)$$

where V_{C0} is the initial cement volume and f_X the initial volume fraction of cement. In the same way, the amount of water remaining is equal to:

$$V_E(t) = V_{E0} - \sum V_E^X \xi_X(t) \text{ avec } V_E^X = V_{C0} \frac{n_E \rho_C f_X / M_X}{n_X \rho_E / M_E} \quad (11)$$

where V_{E0} is the initial volume of water, V_E^X is the volume of water used to hydrate the clinker X and the ratio n_X / n_E refers to the number of water moles n_E consumed to hydrate one mole of clinker n_X with an apparent density of $\rho_C f_X$. The volume of the hydrated elements is computed from the volume of the reactants according to this chemical reaction:



with:

$$V_i^P(t) = \sum_{j=1}^n C_i^j \xi_j(t) \text{ avec } C_i^j = V_{C0} \frac{n_i^R \rho_C f_j / M_j}{n_j^P \rho_i / M_i} \quad (i=1,m) \quad (13)$$

where i and j are respectively the indices associated with products and reactants whose mole number is n_i^R and n_j^P respectively. ρ_i is the product density and M_i its molar mass.

The volume fractions computed from Equation (12) and the mechanical properties of each constituent of the cement paste microstructure provided in literature (Bernard *et al.*, 2003) enable us to simulate the homogenized macroscopic behavior of the cement paste.

3.2. Equivalent homogeneous properties and deformation

Two major stages in the microstructural evolution of cement paste are considered: before the setting, the cement paste is “fluid” and the solid phases are in suspension in water. The capillary porosity (water and gas) of the matrix and the solid phases are chosen as inclusions. After the setting, the cement paste becomes “solid” and we consider a matrix formed by the CSH-phase in which are included the other solid elements (anhydrous cement and hydrates), as well as the pores. The elastic coefficients of each phase have been identified experimentally (Bernard *et al.*, 2003). By including in an iterative way the different inclusions in the matrix according to their volume fraction calculated above, and by using the self-consistent scheme method, the evolution of the homogenized cement paste Young’s modulus can be simulated.

For the calculation of the equivalent strain, the self-consistent scheme method is used, distinguishing pores at different pressures (Lemarchand, 2001). We consider pores containing water and air (whose volume fraction is equal to the sum of volume fractions of water and air) at the capillary pressure and pores containing ettringite at the pressure generated by its formation (Ping and Beaudoin, 1992). The capillary pressure is computed using the following empirical law proposed by Coussy *et al.* (2004):

$$p_c(S) = M(S^{-m} - 1)^{(1-1/m)} \quad (14)$$

where $M = 37.55$ MPa and $m = 2.1648$ are the material parameters and S represents the water saturation rate of the cement paste.

4. Analysis and discussion of the experimental and numerical results

4.1. Application on a cement paste

Figure 1 shows a good correspondence between the results of hydration degree modelling and the experimental values obtained by loss on ignition. Besides, we observe that the evolution of chemical shrinkage is closely related to the progress of cement hydration. Figure 2 provides the simulation results of self-desiccation shrinkage and capillary tension plotted as a function of time. The curve shape indicates that self-desiccation shrinkage directly depends on the capillary tension rate.

Figure 3 shows that the multi-scales modelling enables us to correctly predict the evolution of Young’s modulus after 24 h of hydration. The differences observed between numerical and experimental results between 0 and 24 h can be attributed to

the strong viscous component which characterizes the behaviour of early-age cement paste and which is not taken into account in the present version of the model.

In Figure 4, the comparison between chemical and autogenous shrinkage results as a function of time of hydration highlights the good agreement between simulation and measurement results from the first contact between cement and water up to 24 h. After 24 h, the numerical results underestimate the experimental autogenous shrinkage values. This underestimation could be explained by a « real » capillary tension higher than that calculated with the model. It should also be noted that the numerical values of chemical shrinkage are slightly higher than the experimental values.

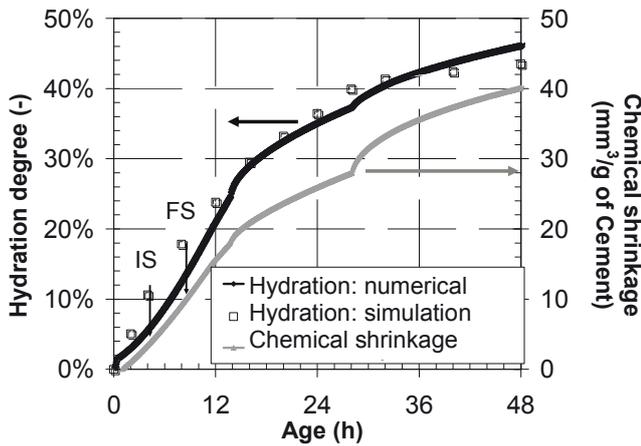


Figure 1. Evolution of hydration degree and chemical shrinkage (IS: initial setting time, FS: final setting time)

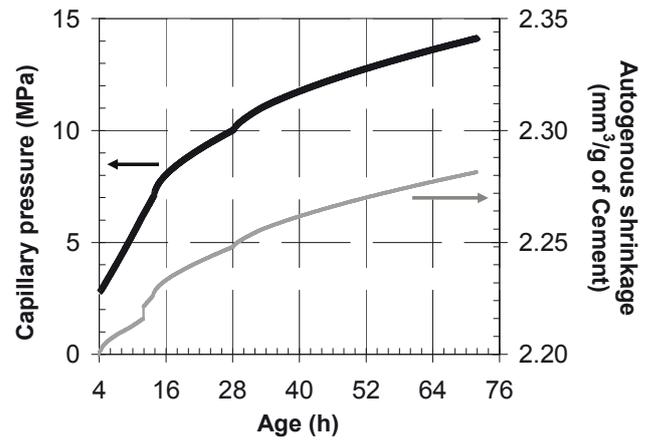


Figure 2. Capillary tension and self-desiccation shrinkage (initialized at setting time)

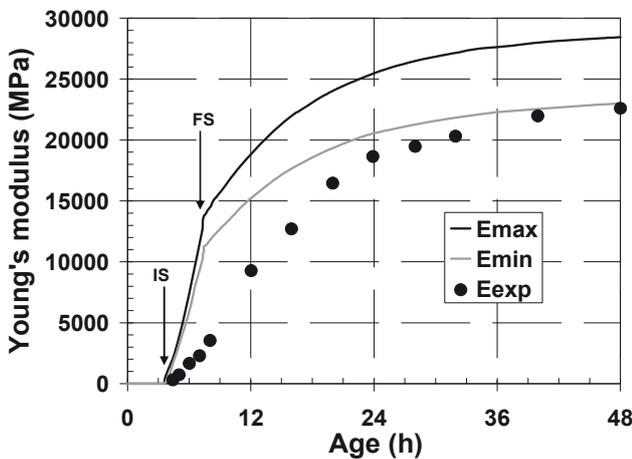


Figure 3. Evolution of Young's modulus (IS: initial setting time, FS: final setting time)

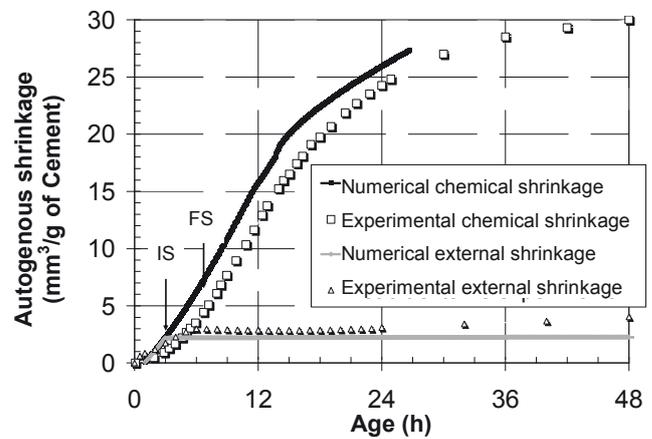


Figure 4. Chemical and autogenous shrinkage (IS: initial setting time, FS: final setting time)

4.2. Cement properties effect

The cement fineness is defined by the cement particle size distribution. Previous experimental research works have shown that an increase in the cement fineness

causes an acceleration of the cement hydration and autogenous shrinkage of cement pastes (Bentz *et al.*, 2007). In the model presented in this paper, the cement fineness is an input parameter for the calculation of hydration. Calculations have been performed to quantify the influence of this parameter on a cement paste with $W/C = 0.4$. The cement composition is given in section 2. Calculated results on the autogenous shrinkage and Young's modulus are plotted in Figure 5. It is observed that the lowest cement fineness gives the highest autogenous shrinkage and Young's modulus values. Indeed, higher cement Blaine fineness induces faster water consumption and therefore more intense capillary tension at 24 h. Besides, cement pastes reach more rapidly their final mechanical properties.

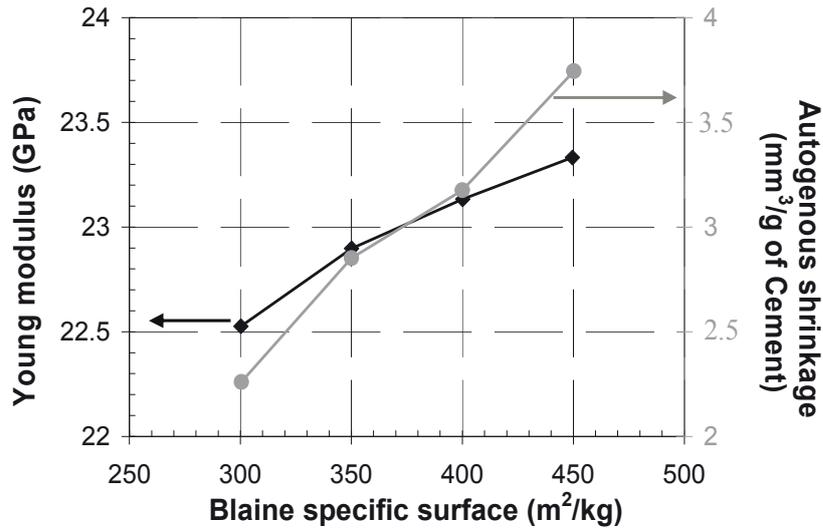


Figure 5. Effect of the Blaine fineness on the autogenous shrinkage and Young's modulus at 24 h of hydration

5. Conclusions

The numerical results presented in this paper demonstrate the capacity of the multi-scales modelling approach to simulate with a certain accuracy the cement hydration rate and the chemical shrinkage evolution. The Young's modulus is correctly estimated after 24 h of hydration. Between 0 and 24 h, the viscoelastic part of the cement paste's behaviour, which is not taken into account in the present version of the model, probably explains the differences between experimental and numerical results. Concerning autogenous shrinkage, simulated and measured values are closed at very early age and diverge after 24 h. Sensitivity tests have shown the influence of the cement fineness on the evolutions of autogenous strain and Young's modulus. Improvements of the model are in progress in order to better simulate the evolution of internal capillary tension and refine the simulation of cement paste autogenous shrinkage at an early stage (≥ 24 h).

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