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Characterization of behavior and cracking of a cement paste confined between spherical aggregate particles

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ABSTRACT

In this work, the cement–grain interactions in a pair of 8 mm spherical grains of quartz or calcite, linked by a Portland cement, are analyzed experimentally at a local scale (cement–grain interface) during mechanical testing. The volume of the cement paste, the water/cement ratio and the gap between the grains are constant. Examined samples are conditioned in an atmosphere of constant temperature (21 °C) and high relative humidity (~100%) to minimize drying shrinkage. At various stages of hydration, the samples are subjected to tensile or compressive tests, monitored by a high-speed camera. Stiffness and force at rupture are estimated and correlated to the crack initiation and propagation followed through image analysis. The results concern mechanical properties and kinetics of cracking in function of hydration time, for both types of loading and both types of grain. Such local characteristics will be used to propose local interaction laws and to support numerical modeling of the concrete at macroscopic scale.

1. Introduction

Concrete is a common component used in construction of buildings, roads, dams, bridges, but also in sensitive objects, as e.g. containment structures of nuclear power plants, where sustainability and stability must be ensured over long periods. The concrete is a granular and heterogeneous material, composed mainly of aggregates (coarse and fine) embedded in a matrix of cement paste (cement mixed with water), which fills the space between the aggregates and glues them together. This material reveals complex mechanical and physical behaviors, depending on its composition and its aging or conditions of its conservation. The concrete passes by a progressive structuration during reactions of hydration of cement, with change of its mechanical properties. Hydration of cement starts immediately after mixing cement with water, and may last during several tens of years [1]. Just after preparation, the concrete is elasto-plastic with pronounced ductility. When hardened, it has a good compressive strength but a low tensile strength and small range of deformation [2–4]. In result, it is very susceptible to cracking, which may have mechanical, physico-chemical or thermal origins. Cracking of the concrete affects significantly its durability, its mechanical parameters and its permeability [1–6]. Cracks in concrete are usually located at the cement–aggregate interface (Interfacial Transition Zone — ITZ), which is often weakened by its increased porosity [7–10]. In order to predict damage and fracture of concrete structures, the knowledge on cement–grain interface behavior has to be improved, with a detailed multi-scale analysis of conditions, origins and mechanisms of cracking. Since macroscopic properties of the concrete depend on their local characteristics, as e.g. physical properties of elementary components, structural composition and micro-geometry of the aggregate–cement interface or transport properties, it is necessary to examine concrete also at the local scale. Local characteristics of concrete and cement are widely described in literature (e.g. [11,12]), often accompanied by mechanical testing at the macroscopic scale, to exhibit relationships between microstructure and mechanical properties of concrete (e.g. [13,14]). To complete these analyses, mechanical tests at the local scale should be also introduced. By now, such tests have attracted only limited attention.

The purpose of this paper is to characterize the behavior and the fracture process of the cement interface at a local scale by experimental analysis of the aggregate–cement matrix interface at different stages of hydration. In particular, the determined mechanical parameters as stiffness $k(j)$ and force at rupture $f(j)$ are correlated with parameters of the kinetics of cracking: the speed of crack growth $v^p(j)$, the rate of crack opening $v^o(j)$, the angle β between the crack and vertical axis of the sample and the tortuosity τ, reconstructed basing on the analysis of high-speed camera images. Characterization of mechanical and physico-chemical interactions in cemented bond will contribute to this knowledge of concrete behavior. It will be also used to

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propose local interaction laws and to support numerical modeling at macro scale.

2. Experiment

In this study, mechanical behavior and cracking of cement interface linking two solid grains are examined at different stages of hydration. Examined samples consist in two identical quartz or calcite spherical grains, linked by a cement paste. Quartz and calcite are the main components of limestone (calcite) and sandstone (quartz) used usually as aggregates (sand, gravels) in concrete production [4].

Each grain has a cylindrical 1.3 mm hole perpendicular to the direction of traction (and compression), which allows to fix the sample during tensile testing. The cement paste is prepared using Portland cement and distilled water, with a water–cement ratio (W/C) of 0.5. The geometric configuration of the sample is shown in Fig. 1, while "material" data are given in Table 1.

The samples are prepared using PVC molds, with constant gap between the grains and constant mass (and volume) of the cement paste. After preparation, samples are conditioned in stable and controlled atmosphere, with temperature of 21 °C and high relative humidity (~100%). Such conditions limit the evaporation from boundary surfaces and therefore minimize the drying shrinkage. Samples are produced in series of about 10 each day, which gives a possibility to examine the samples at each stage of hydration, with several repetitions of each measurement.

Before mechanical testing, the PVC molds are removed and each sample is weighed and measured. At this stage, the samples which present some visible defects, as gaps, excessive deformation or initial cracking, are eliminated. This procedure allows to select only "well-done" samples to increase the quality and the repeatability of the results. Qualified samples are than fixed to the support of the MTS Universal Testing Machine M1/E [15], in order to carry out the mechanical tests (compressive and tensile testing).

Mechanical tests are realized mainly on early-age samples (from 1 to 28 days of hydration) at constant loading speed of 0.01 mm/s. During compression tests, the sample is placed between two plates, while during tensile testing the sample is fixed with pins inserted in the cylindrical holes (see Fig. 1b). For each test, the force–displacement curve is recorded using testwork4 software [15] and then post-processed using MS Excel and SciDAVis software [16]. Analysis of these curves allows to determine the peak force at rupture (maximal force) $f(j)$ and stiffness $k(j)$. The onset of cracking and rupture of the sample is recorded with use of a high-speed digital camera (Vision Research Phantom v12 [17]) at 40,000 frames per second. Obtained image sequences are treated using the ImageJ and MBRuler Pro softwares [18,19] in order to estimate the evolution of some geometrical parameters in function of hydration time j and crack progression time t, such as sample diameter $d(z, t)$, crack opening $c(z, t)$, crack length $l_T(j)$, $l_p(j)$ (see Fig. 1c) or angle β between the crack and the vertical axis of the sample (Fig. 1c).

Table 1
Material data of used components.

Grains		Cement paste	
Diameter of grains	8.26 ± 0.09 mm	Formula	CEM II/B-LL 32.5 N + distilled water
Calcite: formula	$CaCO_3$		
Calcite: density	2.6 g/cm^3	W/C ratio	0.5
Quartz: formula	SiO_2	Mass	0.5 g/sample
Quartz: density	2.65 g/cm^3	Density (measured)	2.22 ± 0.22 g/cm^3

These variables are used to reconstruct some parameters related to the kinetics of cracking, as time $t_p(j)$ at which the crack length is maximal, elapsed since the crack onset, the maximum of crack propagation speed $v^p(j)$ (Eq. (1)), the maximum of crack opening rate $v^o(j)$ (Eq. (2)) and crack tortuosity $\tau(j)$ (Eq. (3)).

$$v^p(j) = \delta l_T(j)/\delta t \tag{1}$$

$$v^o(j) = \delta c(j)/\delta t \tag{2}$$

$$\tau(j) = l_T(j)/l_p(j) \tag{3}$$

3. Results

Observations of the early-age samples before mechanical tests reveal that the apparent color of the surface, the volume fraction and the size of visible hydrates depend on the hydration time (see Fig. 2). Development of hydrates as function of hydration time is a well-known result of chemical reactions of hydration, described widely by many authors [3–5].

3.1. Compression tests

For each compression test, force–displacement curves were recorded, as shown in Fig. 3. In general, one can observe a significant increase both of the maximal force $f_C(j)$ and of the stiffness of the samples $k_C(j)$ at the beginning of hydration. The stiffness $k_C(j)$ remains constant after about 10 days of hydration, while the maximal force $f_C(j)$ remains constant after more than 3 weeks of hydration.

A gentle decrease of the force after rupture is observed for the samples at very early-age. After several days of hydration, the samples become more brittle and a sudden force jump is observed just after the rupture (see Fig. 3). In general, the behavior of both types of samples is similar. Nevertheless, some differences are observed on force–displacement curves for early-age samples (first week) and they are not explained yet.

The evolution of maximum force $f_C(j)$ as a function of hydration time is presented in Fig. 4a, for both types of samples. The force at rupture $f_C(j)$ during compression tests is rather small at initial hydration stage

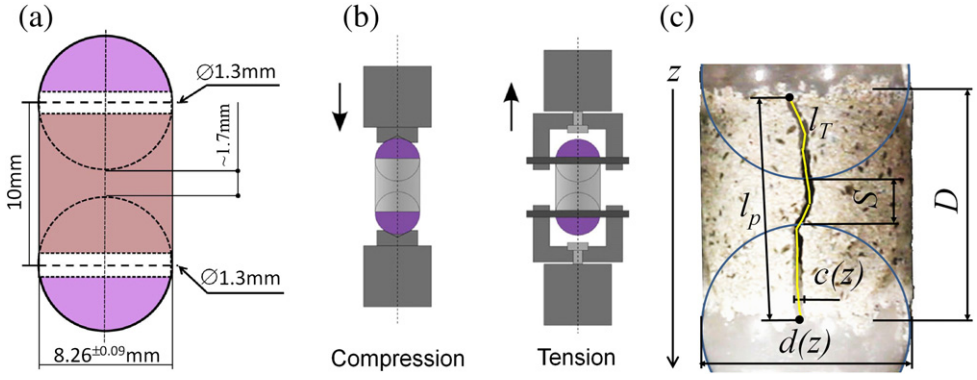

Fig. 1. Dimensions of the sample (a), supports for mechanical testing (b), and measured geometrical parameters (c).