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Long-term creep properties of cementitious materials: Comparing microindentation testing with macroscopic uniaxial compressive testing

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ABSTRACT

This study is dedicated to comparing minutes-long microindentation creep experiments on cement paste with years-long macroscopic creep experiments on concrete and months-long macroscopic creep experiments on cement paste. For all experiments, after a transient period the creep function was well captured by a logarithmic function of time, the amplitude of which is governed by a so-called creep modulus. The non-logarithmic transient periods lasted for days at the macroscopic scale, but only for seconds at the scale of microindentation. The creep moduli (which thus govern the rate of the long-term logarithmic creep) of concrete samples were estimated from microindentations performed at the scale of cement pastes in combination with micro-mechanical models. Those estimates were proportional to the creep moduli measured on concrete samples by regular macroscopic uniaxial testing, thus proving that minutes-long microindentation can provide a measurement of the long-term creep properties of cementitious materials.

1. Introduction

In concrete, a variety of phenomena can lead to deformations that evolve over time: autogenous shrinkage, drying shrinkage, aging… Out of this variety, one phenomenon is basic creep, which is defined as the time-dependent deformation that is only due to the application of an external mechanical load. In this study, we focus on this basic creep (samples were sealed to prevent any desiccation). Creep of concrete is usually divided into at least two distinct kinetics [4]: a short-term creep, followed by a deviatoric long-term creep.

Indeed, concrete creeps, i.e., slowly deforms over time when subjected to constant stress. Both short-term and long-term creeps are important for the stability, durability, and serviceability of concrete structure. The importance of taking creep deformation into consideration in the design of concrete structures was recalled recently [3]. The deformation due to creep evolves over years or even over decades. Therefore, in order to get a reliable prediction of the long-term creep deformations of concrete, various authors recommend for creep experiments on concrete to last for at least several months [15,16]. The long duration of those experiments makes it not only time-consuming but also difficult to characterize creep properties. Indeed, over those long periods of time, experimental parameters must be very well controlled: for instance, the load has to remain constant, temperature must not vary and hydric exchanges with the surroundings must be prevented. In addition, since other physical phenomena can lead to time-dependent deformations of the concrete samples, basic creep is usually measured by performing two experiments in parallel [16]: deformations due to basic creep are calculated as the difference between the time-dependent deformation of a sealed sample under load and the time-dependent deformation of another sealed sample without external load (autogenous shrinkage). This need to run two experiments in parallel increases experimental uncertainties, so that a dispersion of about 16.5% on long-term creep results on concrete samples loaded at 28 days can eventually be expected [5]. For samples loaded at an early age, this dispersion is rather on the order of 20% [5].

The creep of concrete is mainly due to the creep of cement paste [12]. For Portland cement, its creep behavior is mainly determined by its porosity and the creep properties of C–S–H (i.e., of calcium silicate hydrates). In order to measure mechanical properties of individual phases of heterogeneous materials, the indentation technique proves to be well-suited [6,7,30,36,20,21]. The possibility to measure viscous properties by indentation in particular has been shown on polymers [23,18], metals [27], cementitious materials [33,8,34,24], and so on. Therefore, in order to predict the creep behavior of concrete, one could think of characterizing the creep behavior of cement paste, or of C–S–H, and upscale this behavior to the scale of concrete samples. Vandamme and Ulm showed that the long-term kinetics of concrete can indeed be quantitatively estimated from a grid of nanoindentation tests performed at the sub-micrometer scale of the C–S–H phases [34].

In the present work, we aim at verifying whether an estimation of the macroscopic creep behavior of concrete samples can be inferred from microindentation tests performed at the scale of the cement

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paste. With this objective, we compared minutes-long microindentation creep experiments on cement paste samples with months-long macroscopic uniaxial creep experiments on cement paste samples and years-long macroscopic uniaxial creep experiments on concrete samples. The next section is dedicated to describing the materials and methods. Results are then presented and discussed, before conclusions are drawn.

2. Materials and methods

Both cement samples and concrete samples were prepared. On the concrete samples, years-long macroscopic uniaxial creep experiments were performed. On the cement samples, both months-long macroscopic uniaxial creep experiments and minutes-long microindentation creep experiments were performed.

2.1. Materials

All cement paste samples and concrete samples were made with Portland cement (class CEM I 52.5). Both clinkers from Saint Vigor (Lafarge, France) and from Saint-Pierre-la-Cour (Lafarge, France) were used, which contain different amounts of tricalcium aluminate (see Table 1). Concrete samples and cement samples for uniaxial creep testing were manufactured in 1992 in the framework of Le Roy's Ph.D. thesis [16], while cement samples for microindentation creep testing were manufactured in 2011 in the framework of Zhang's Ph.D. thesis. Clinkers from Saint Vigor used in 1992 and in 2011 were from the same factory, as was the case for clinkers from Saint-Pierre-la-Cour. The composition of the clinkers used in the various samples is provided in Table 1, while their physical properties are provided in Table 2. Although the clinkers used in the samples for uniaxial testing and for microindentation testing were manufactured about 20 years apart, the composition and the specific gravity of the two batches differed very little from each other. The proportion of the main phases in the clinkers used to prepare cement pastes for microindentation testing is given in Table 3.

In some samples, silica fume was used as an additive. Both silica fume used in 1992 and in 2011 were from Laudun (France). As can be observed in Table 1, from one set to the other the content of SiO_2 varied by about 6%. Also, Table 2 shows that the specific gravity of the silica fume used in 2011 was about 20% greater than that of the silica fume used in 1992. To some samples a superplasticizer was added (see Table 4), the solid content of which was 30.5% and the effective component of which was melamine.

The mix formulation of the various samples used in this study is given in Table 4. Cylindrical concrete samples were prepared in 1992 with seven various mix formulations. For each formulation, four samples were dedicated to uniaxial strength testing (the diameter of these samples was 110 mm and their height was 220 mm), one sample was dedicated to autogenous shrinkage testing (the diameter of this sample was 160 mm and its height was 1000 mm), and the last sample was dedicated to uniaxial creep testing (the geometry of this sample was the same as that of the sample dedicated to autogenous shrinkage). The mix formulations of those concretes differed by the water-to-cement ratio w/c, the mass ratio s/c of silica fume to clinker, and the volume fraction of aggregates (i.e., of sand and gravel).

Table 1
Mass percentage of chemical components in the clinkers and silica fume used in this study. Data is provided by the manufacturer (Lafarge). For clinker and silica fume, respectively, only mass percentages greater than 1% and than 3% are given.

	Year	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	LOI[a]
Cement from Saint Vigor	1992	64.25	22.49	3.60	4.00	2.50	1.48
	2011	64.76	20.87	3.58	4.45	2.45	1.06
Cement from Saint-Pierre-la-Cour	1992	65.30	19.72	4.98	2.71	3.36	1.30
	2011	63.94	20.06	4.93	2.86	3.67	1.45
Silica fume from Laudun	1992	–	87.00	–	–	–	3.09
	2011	–	93.31	–	–	–	3.43

[a] LOI: loss on ignition.

Table 2
Physical properties of clinker and silica fume used in this study. Data is provided by the manufacturer (Lafarge).

	Year	Specific surface ($m^2 \cdot g^{-1}$)	Specific gravity ($g \cdot cm^{-3}$)
Cement from Saint Vigor	1992	0.35	3.17
	2011	0.35	3.18
Cement from Saint-Pierre-la-Cour	1992	–	–
	2011	0.45	3.11
Silica fume from Laudun	1992	17.6	2.20
	2011	21.3	–

Six groups of cylindrical cement paste samples were prepared in 1992 with a diameter equal to 20 mm and a height equal to 160 mm. For each group, two samples were prepared: one was used for autogenous shrinkage test and the other one for uniaxial creep test. The mix formulations of those pastes differed by the water-to-cement ratio w/c, the mass ratio s/c of silica fume to clinker, and the type of clinker used (from Saint Vigor or from Saint-Pierre-la-Cour). Samples with identical mix formulations and geometry were prepared again in 2011 for microindentation creep test. In addition, the cement paste P33-1SV (see Table 4 for sample designation) was also prepared for microindentation testing, although no paste with this mix formulation was tested by uniaxial compression: by doing so, all cement pastes used in both cement pastes and concretes in 1992 were manufactured again in 2011 for microindentation testing.

Samples were prepared according to the following procedure. For cement paste samples the mixing consisted in: adding the solid raw materials, the water, and one third of the superplasticizer; mixing for 3 min; adding the rest of the superplasticizer; mixing for 2 min. For concrete samples the mixing consisted in: adding the solid raw materials; mixing for 1 min; adding water and one third of the superplasticizer; mixing for 2 min; adding the rest of the superplasticizer; mixing for 1 min. After molding, for both cement paste samples and concrete samples, embedded gas bubbles were evacuated by vibration on a vibration table; samples were unmolded 24 h after mixing and enveloped in 2 layers of self-sealing aluminum paper; samples were conserved at 20 °C ± 1 °C and at a relative humidity 50% ± 5% till testing. For cement paste only, right after vibration the samples were rotated for 15 h in order to prevent any segregation.

2.2. Years-long uniaxial compression creep experiments on concrete

On the concrete samples, basic creep was measured up to 15 years. This basic creep was obtained by performing in parallel autogenous shrinkage test on one sample and creep test on another sample with identical mix formulation and geometry. The autogenous shrinkage test started 24 h after casting. During this test, no load was applied to the sample and the axial strain $\epsilon_s(t)$ was measured over time. On the samples to be loaded for the creep experiments by uniaxial compression, we also started measuring a total axial strain $\epsilon_t(t)$ 24 h after casting. On these samples, the application of a uniaxial compression started 28 days after casting. During the creep periods, a uniaxial compressive stress σ_u

Table 3
Proportion of the main phases in the clinkers used in 2011 to prepare cement paste samples for microindentation testing, determined by Rietveld X-ray diffraction quantification. Data is provided by the manufacturer (Lafarge).

Cement	C_3S	C_2S	C_3A	C_4AF	Gypsum
Saint Vigor (2011)	60.0	22.4	1.20	12.9	1.30
Saint-Pierre-la-Cour (2011)	59.9	17.6	7.40	9.40	0.30