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New evidences on the effect of the internal relative humidity on the creep and relaxation behaviour of a cement paste by micro-indentation techniques

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1. Introduction

A recent survey on several concrete bridges monitored over a period of more than 20 years has showed that creep deflections exceed by far the predictions of several national codes, launching a wake-up call on the importance of better understanding and characterizing long term creep of concrete [1,2]. Estimating longterm creep of concrete using methods based on short term tests has been unavoidable and yet unsolved crux in civil engineering [3]. The difficulty of predicting concrete creep may be explained by the complex mechanisms at stake, e.g.: (i) micro-diffusion of water molecules between hindered adsorbed layers and capillary pores [4,5]; (ii) slip mechanisms along interfaces of Calcium-Silicate-Hydrates (C-S-H) sheets [6], like a viscous flow in shear sites which are responsible relaxation of microprestress in the micropores of cementitious materials [7]; (iii) re-arrangement of cement paste gel like a compaction [8,9];(iv) micro-cracking interacting with microdiffusion [10].

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ABSTRACT

A recent survey has revealed that the deflection of concrete bridges increases incessantly over a period of more than 20years and can exceed by far the predictions of current design recommendations. This work applies microindentation techniques, which allow quickly assessing the long term rate, to investigate the effect of relative humidities on both the viscous behaviour of a cement paste. A large campaign of microindentation tests was carried out at different relative humidities, loading histories, curing times, and load levels for both creep and relaxation testing. The correlations between creep rate, relaxation rate, and relative humidity were investigated. The results showed that the increase of the relative humidity reduces the indentation modulus and hardness, but increases the long term creep rate of a cement paste. The effect of the relative humidity on the creep rate was possibly explained by local forces acting on Calcium-Silicate-Hydrates sheets or by microcracking drying effects.

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It is today widely recognized that short term creep is largely affected by stress-induced water movements and the redistribution in the capillary porous space [11–14], while long term creep is due to the sliding of layered structures within the C-S-H gels [7,15,16]. Basic creep which by definition does not imply water exchange, is well recognized to have a logarithmic dependence on time in the long term [1,17-19].

The water in content pores has a strong effect on the timedependent deformation of concrete [20]. A pre-dried concrete creeps little or not at all, but its creep capacity can be restored by rewetting [21,22]. The few results available in open literature on the concrete creep at long term are somehow contradictory. Troxell et al. [19] performed compressive tests on concrete samples (with a diameter varying from 100 to 250 mm) for a period of about 23 years by varying the RH of storage. He observed that after 1 year, creep was rather logarithmic in time, but independent of the RH of storage. In another landmark work, Brooks carried a wide campaign on concrete creep for a period of 30 years at about 70 ° C [23] by varying waterto-cement ratios, aggregate types, stored dry (RH = 65%) and stored wet conditions. The creep deformation appeared rather logarithmic in time after about 1 year and its creep rate was greater for the wet stored samples. Even more, the greater the water-to-cement ratio

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was (i.e. the water content in the pores), the greater the creep rate resulted. Furthermore, Ruetz [11] performed creep tests at hygral equilibrium before testing confirming that long term basic creep increases considerably when the content of the evaporable water is greater. In spite of the important effect, few experimental studies on the effect of RH on long term creep are available in open literature.

Such apparent incoherency in the aforementioned results may be due to the long time needed for reaching a uniform distribution of the moisture content within a concrete sample, e.g., several years for a sample with diameter of few centimeters. Therefore, unavoidable RH gradients are present and the apparent macroscopic creep may not be representative of the true creep at the material level [5,24]. Few works employed samples of small size to favour a uniform RH distribution at hygral equilibrium. For instance, Wittman carried out compressible creep tests on hollow cylinders of few millimeters size brought to hygral equilibrium at different RH prior to applying the sustained load showing that the lower the pore humidity, the smaller is the creep rate, provided that the specimen is in hygral equilibrium [25]. Based on those results, Bazant proposed that creep rate of cement paste increases in a parabolic manner with the RH extent [5]. Finally, Alizadeh et al. [26] carried out relaxation bending test on small beams with a span of about 60 mm made of synthetic C-S-H showing that the relaxation rate increases when RH increases.

In the last decade, micro-indentation techniques have provided a means to assess the long term creep rate behaviour of cement pastes in a very short time thanks to the tiny probed volume which is of few micrometer size [27,28]. In more details, the logarithmic creep rate measured by microindentation after about 300 s was found to fairly well correlate the logarithmic creep rate under compressive loading at about 30 years for several kinds of concretes [29]. Considering the average half-time water diffusion of a cement paste, one can approximately estimate that water takes few dozens of seconds to diffuse through the few hundreds of micrometers size which corresponds to the volume size typically probed by a microindentation [24,30]. As the short term creep mechanisms due to water diffusion exhaust in few seconds, it is then possible to assess the logarithmic long term creep rate due to the C-S-H sliding [6]. Furthermore, microindentation appears a convenient means to rapidly achieve a uniform moisture distribution within the micrometer size of material under testing. Lately, Zhang found that the RH increases long term creep rate measured by microindentation for synthetic C-S-H by a factor of 5 when the RH level increases from 11% to 95% [31].

The scope of this work is to further apply microindentation techniques to better understand the effect of humidity on both creep and relaxation behaviour of cement paste. In particular, a wide experimental campaign was carried out by varying several test settings (e.g., curing condition, maximum loading, holding time), showing a fairly satisfactory repeatability in test results. Finally, correlation between creep and relaxation rates at different RH was studied and discussed to better understand the creep mechanisms.

2. Methods and materials

2.1. Basics of microindentation techniques

In the last decade, instrumented indentation techniques have been successfully applied to measure the mechanical properties of cementitious materials [32,33]. As sketched in Fig. 1, an indentation test consists of penetrating the flat surface of a material with a rigid diamond indenter by applying an external load *P* and simultaneously measuring the penetration depth *h*. Fig. 2e shows a typical *P*(*h*) curve with the maximum load (P_{max}). For this work, we employed a Berkovich indenter, which is three-sided pyramid with an effective conical shape angle (α) of 70.3°.



Fig. 1. Schematic view of an indentation test with a conical tip.

An indentation test can be performed either in load control or displacement control as schematically represented in Fig. 2. Typically in a force (or displacement) control tests the external load P (or the displacement h) is applied in 3 phases as shown in Fig. 2a (or Fig. 2b), such as: (i) the loading phase: the load P_{max} (or the displacement $h_{\rm max}$) is linearly applied in a time $\tau_{\rm I}$. Note that the corresponding curve P(h) is parabolic for a conical indenter as the contact area is increasing during loading [34]; (ii) the holding phase: the load P_{max} (or the displacement h_{max}) is kept constant for a time τ_{H} . If the material is viscous, the indentation depth creeps as shown in Fig. 2c (or the load relaxes as shown in Fig. 2d); (iii) the unloading phase: the load P_{max} (or the displacement h_{max}) is released within a time τ_U . The latter is supposed to be elastic and gives access to the elastic property of the material trough the slope S of Fig. 2e or f. Upon load release, a residual penetration depth is often observed due to plasticity. Displacement control tests present some advantages with respect to load control tests, such as: (i) no delayed inelastic deformation (e.g., plasticity or cracking) can occur during holding phase [29]; (ii) contact area A_c is a known constant in the holding phase; (iii) evolution of the hardness H(t) during the holding phase is known. Generally, load is applied much quicker ($\tau_L = 5$ s) than the holding time (τ_L =300 s) in order to observe creep deformation during holding phase [27,31,35].

The load-displacement curve P(h) is then usually analysed by a continuum model [36,37] to assess the indentation properties. First, the indentation modulus M is estimated from the slope S = dP/dh at the initial stage of the unloading branch of the curve P(h) as follows:

$$M = \frac{S\sqrt{\pi}}{2\beta\sqrt{A_c}} = \frac{S}{2\beta a_u} \tag{1}$$

where $A_c = \pi a_u^2$ is the projected contact area between the indenter and the substrate and β coefficient accounts for the slip on the indenter surface, which is about 1.034 for the Berkovich-type indenter [37]. The indentation hardness *H* is equivalent to the mean pressure supported by the sample under maximum load and it defined as follows:

$$H = \frac{P_{\text{max}}}{A_c} \tag{2}$$

The material properties need be derived from the indentation properties upon the choice of a material model. For instance, the Young's modulus E of an elastic isotropic homogeneous material is estimated from the indentation modulus M as follows:

$$M^{-1} = \frac{1 - \nu_i^2}{E_i} + \frac{1 - \nu^2}{E}$$
(3)

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