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1 Long-term Creep Properties of Cementitious Materials:
2 Comparing Microindentation Testing with Macroscopic
3 Uniaxial Compressive Testing

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9 **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.

10 *Keywords:* Creep, Long-Term Performance, Mechanical Properties

11 **1. Introduction**

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

20 Indeed, concrete creeps, i.e., slowly deforms over time when subjected to
21 constant stress. Both short term and long term creep are important for the
22 stability, durability, and serviceability of concrete structure. The importance
23 of taking creep deformation into consideration in the design of concrete struc-
24 tures was recalled recently [3]. The deformation due to creep evolves over
25 years or even over decades. Therefore, in order to get a reliable prediction
26 of the long-term creep deformations of concrete, various authors recommend
27 for creep experiments on concrete to last for at least several months [15, 16].
28 The long duration of those experiments makes it not only time-consuming but
29 also difficult to characterize creep properties. Indeed, over those long periods
30 of time, experimental parameters must be very well controlled: for instance,
31 the load has to remain constant, temperature must not vary and hydric ex-
32 changes with the surroundings must be prevented. In addition, since other
33 physical phenomena can lead to time-dependent deformations of the concrete
34 samples, basic creep is usually measured by performing two experiments in
35 parallel [16]: deformations due to basic creep are calculated as the difference

36 between the time-dependent deformation of a sealed sample under load and
37 the time-dependent deformation of another sealed sample without external
38 load (autogenous shrinkage). This need to run two experiments in parallel
39 increases experimental uncertainties, so that a dispersion of about **16.5% on**
40 **long-term creep results on concrete samples loaded at 28 days** can eventually
41 be expected [5]. **For samples loaded at an early age, this dispersion is rather**
42 **on the order of 20% [5].**

43 The creep of concrete is mainly due to the creep of cement paste [12].
44 For Portland cement, its creep behavior is mainly determined by its porosity
45 and the creep properties of C-S-H (i.e., of calcium silicate hydrates). In
46 order to measure mechanical properties of individual phases of heterogeneous
47 materials, the indentation technique proves to be well-suited [6, 7, 30, 36,
48 20, 21]. The possibility to measure viscous properties by indentation in
49 particular has been shown on polymers [23, 18], metals [27], cementitious
50 materials [33, 8, 34, 24], and so on. Therefore, in order to predict the creep
51 behavior of concrete, one could think of characterizing the creep behavior of
52 cement paste, or of C-S-H, and upscale this behavior to the scale of concrete
53 samples. Vandamme and Ulm showed that the long-term kinetics of concrete
54 can indeed be quantitatively estimated from a grid of nanoindentation tests
55 performed at the sub-micrometer scale of the C-S-H phases [34].

56 In the present work, we aim at verifying whether an estimation of the
57 macroscopic creep behavior of concrete samples can be inferred from mi-
58 croindentation tests performed at the scale of the cement paste. With this
59 objective, we compared minutes-long microindentation creep experiments on
60 cement paste samples with months-long macroscopic uniaxial creep experi-

61 ments on cement paste samples and years-long macroscopic uniaxial creep
62 experiments on concrete samples. The next section is dedicated to describing
63 the materials and methods. Results are then presented and discussed, before
64 conclusions are drawn.

65 **2. Materials and methods**

66 Both cement samples and concrete samples were prepared. On the con-
67 crete samples, years-long macroscopic uniaxial creep experiments were per-
68 formed. On the cement samples, both months-long macroscopic uniaxial
69 creep experiments and minutes-long microindentation creep experiments were
70 performed.

71 *2.1. Materials*

72 All cement paste samples and concrete samples were made with Portland
73 cement (class CEM I 52.5). Both clinkers from Saint Vigor (Lafarge, France)
74 and from Saint-Pierre-la-Cour (Lafarge, France) were used, which contain
75 different amounts of tricalcium aluminate (see Table 1). Concrete samples
76 and cement samples for uniaxial creep testing were manufactured in 1992,
77 while cement samples for microindentation creep testing were manufactured
78 in 2011. Clinkers from Saint Vigor used in 1992 and in 2011 were from the
79 same factory, as was the case for clinkers from Saint-Pierre-la-Cour. The
80 composition of the clinkers used in the various samples is provided in Table
81 1, while their physical properties are provided in Table 2. Although the
82 clinkers used in the samples for uniaxial testing and for microindentation
83 testing were manufactured about 20 years apart, the composition and the
84 specific gravity of the two batches differed very little from each other. The

	Year	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O 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

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.

(a) LOI: loss on ignition.

85 proportion of the main phases in the clinkers used to prepare cement pastes
86 for microindentation testing is given in Table 3.

87 In some samples, silica fume was used as an additive. Both silica fume
88 used in 1992 and in 2011 were from Laudun (France). As can be observed
89 in Table 1, from one set to the other the content of SiO₂ varied by about
90 6%. And Table 2 shows that the specific gravity of the silica fume used in
91 2011 was about 20% greater than that of the silica fume used in 1992. To
92 some samples a superplasticizer was added (see Table 4), the solid content
93 of which was 30.5% and the effective component of which was melamine.

94 The mix formulation of the various samples used in this study is given
95 in Table 4. Cylindrical concrete samples were prepared in 1992 with seven
96 various mix formulations. For each formulation, four samples were dedicated
97 to uniaxial strength testing (the diameter of these samples was 110 mm and
98 their height was 220 mm), one sample was dedicated to autogenous shrinkage
99 testing (the diameter of this sample was 160 mm and its height was 1000 mm),

	Year	Specific surface ($\text{m}^2.\text{g}^{-1}$)	Specific gravity ($\text{g}.\text{cm}^{-3}$)
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	-

Table 2: Physical properties of clinker and silica fume used in this study. 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

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).

100 and the last sample was dedicated to uniaxial creep testing the geometry of
101 this sample was the same as that of the sample dedicated to autogenous
102 shrinkage). The mix formulations of those concretes differed by the water-
103 to-cement ratio w/c , the mass ratio s/c of silica fume to clinker, and the
104 volume fraction of aggregates (i.e., of sand and gravel).

105 Six groups of cylindrical cement paste samples were prepared in 1992
106 with a diameter equal to 20 mm and a height equal to 160 mm. For each
107 group, two samples were prepared: one was used for autogenous shrinkage
108 test and the other one for uniaxial creep test. The mix formulations of
109 those pastes differed by the water-to-cement ratio w/c , the mass ratio s/c
110 of silica fume to clinker, and the type of clinker used (from Saint Vigor or
111 from Saint-Pierre-la-Cour). Samples with identical mix formulations and
112 geometry were prepared again in 2011 for microindentation creep test. In
113 addition, the cement paste P33-1SV (see Table 4 for sample designation)
114 was also prepared for microindentation testing, although paste with this mix
115 formulation was not tested by uniaxial test: by doing so, all cement pastes
116 used in both cement pastes and concretes in 1992 were manufactured again
117 in 2011 for microindentation test.

118 Samples were prepared according to the following procedure. For cement
119 paste samples the mixing consisted in: adding the solid raw materials, the
120 water, and one third of the superplasticizer; mixing for 3 minutes; adding
121 the rest of superplasticizer; mixing for 2 minutes. For concrete samples the
122 mixing consisted in: adding the solid raw materials; mixing for 1 minute;
123 adding water and one third of the superplasticizer; mixing for 2 minutes;
124 adding the rest of the superplasticizer; mixing for 1 minute. After molding,

Sample	Cement	$w/c^{(a)}$	$s/c^{(b)}$	$p/c^{(c)}$	$f_{agg}^{(d)}$ (%)	1992 ^(e)	2011 ^(f)
P28-0SV	Saint Vigor	0.28	0.0	1.5%	-	X	X
P38-0SV	Saint Vigor	0.38	0.0	0.0%	-	X	X
P50-0SV	Saint Vigor	0.50	0.0	0.0%	-	X	X
P28-1SV	Saint Vigor	0.28	0.1	1.5%	-	X	X
P33-1SV	Saint Vigor	0.33	0.1	1.5%	-		X
P38-1SV	Saint Vigor	0.38	0.1	1.5%	-	X	X
P38-0LC	Saint-Pierre -la-Cour	0.38	0.0	1.5	-	X	X
B28-1	Saint Vigor	0.28	0.1	4.7%	71.0	X	
B38-1	Saint Vigor	0.38	0.1	4.9%	71.3	X	
B50-0	Saint Vigor	0.50	0.0	0.0%	70.5	X	
B33-1A	Saint Vigor	0.33	0.1	4.9%	67.3	X	
B33-1B	Saint Vigor	0.33	0.1	4.9%	69.5	X	
B33-1C	Saint Vigor	0.33	0.1	4.8%	71.5	X	
B33-1D	Saint Vigor	0.33	0.1	4.9%	73.1	X	

Table 4: Mix formulations of cement paste samples (the denomination of which starts with the letter P) and concrete samples (the denomination of which starts with the letter B) prepared in this study.

(a) w/c denotes the water-to-cement mass ratio; (b) s/c stands for the mass ratio of silica fume to clinker; (c) p/c denotes the superplasticizer to cement ratio; (d) f_{agg} denotes the volume fraction of aggregates (i.e., gravel and sand) in concrete; (e) samples prepared in 1992 were used for uniaxial creep experiments; (f) samples prepared in 2011 were used for microindentation creep experiments.

125 for both cement paste samples and concrete samples, embedded gas bubbles
126 were evacuated by vibration on a vibration table; samples were unmolded 24
127 hours after mixing and enveloped in 2 layers of self-sealing aluminum paper;
128 samples were conserved at $20^{\circ}\text{C} \pm 1^{\circ}\text{C}$ and at a relative humidity $50\% \pm 5\%$
129 till testing. For cement paste only, right after vibration the samples were
130 rotated for 15 hours in order to prevent any segregation.

131 *2.2. Years-long uniaxial compression creep experiments on concrete*

132 On the concrete samples, basic creep was measured up to 15 years. This
133 basic creep was obtained by performing in parallel autogenous shrinkage
134 test on one sample and creep test on another sample with identical mix
135 formulation and geometry. The autogenous shrinkage test started 24 hours
136 after casting. During this test, no load was applied to the sample and the
137 axial strain $\epsilon_s(t)$ was measured over time. **On the samples to be loaded for the**
138 **creep experiments by uniaxial compression, we also started measuring a total**
139 **axial strain $\epsilon_t(t)$ 24 hours after casting. On these samples, the application**
140 **of a uniaxial compression** started 28 days after casting. During **the creep**
141 **periods**, a uniaxial compressive stress σ_u equal to 30% of the 28-day uniaxial
142 compression strength was applied and kept constant, and **we kept measuring**
143 the axial strain $\epsilon_t(t)$ **over** time. The reference time $t = 0$ corresponds to
144 the time at which the load was applied for the creep experiments, i.e., to
145 28 days after casting. The compression strength was obtained on a distinct
146 sample with the same mix formulation and the same geometry just before the
147 commencement of the creep test, by following the then-used French standard
148 NFP 18-406. The duration of the tests varied from 150 days to 5230 days
149 (i.e., about 14.5 years) for the various samples. All tests were performed in

150 sealed conditions at $20^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

151 For a linear viscoelastic sample subjected to a known uniaxial stress $\sigma_u(t)$
152 applied over positive time $t > 0$, the resulting uniaxial strain $\epsilon_u(t)$ can be
153 calculated through the uniaxial creep compliance $J_u(t)$ with [26]:

$$\epsilon_u(t) = \int_0^t J_u(t - \tau) \dot{\sigma}_u(\tau) d\tau \quad (1)$$

154 where \dot{f} stands for the time derivative of a function f . At time $t = 0$, the
155 uniaxial creep compliance must be equal to: $J_u(t = 0) = 1/E_0$, where E_0 is
156 the elastic Young's modulus of the material. The function $J_u(t) - J_u(0) =$
157 $J_u(t) - 1/E_0$ is known as the uniaxial creep function.

158 For mature concrete subjected to negligible variations of temperatures,
159 linear viscoelasticity is expected to apply reasonably well, as long as the
160 applied stresses increase or slightly decrease over time [13]. For such materi-
161 als, which can also be subjected to drying-induced shrinkage or autogenous
162 shrinkage, the correct strain to consider in Eq. (1) is the so-called basic creep
163 strain $\epsilon_b(t)$. From the uniaxial experiments here performed on concrete sam-
164 ples, this basic creep strain $\epsilon_b(t)$ was obtained as the difference between the
165 total axial strain $\epsilon_t(t)$ measured on the concrete sample under load and the
166 axial strain $\epsilon_s(t)$ due to autogenous shrinkage and measured on the concrete
167 sample subjected to no load: $\epsilon_b(t) = \epsilon_t(t) - \epsilon_s(t)$. Since the load was kept
168 constant over time during the macroscopic creep experiments, a direct use of
169 Eq. (1) shows that the uniaxial basic creep compliance $J_u(t)$ of the concrete
170 samples could be obtained with the following formula:

$$J_u(t) = \frac{\epsilon_b(t)}{\sigma_u} = \frac{\epsilon_t(t) - \epsilon_s(t)}{\sigma_u} \quad (2)$$

171 We recall that the function $J_u(t) - J_u(0) = J_u(t) - 1/E_0$ is the uni-
172 axial creep function. The creep experiments started 28 days after casting,
173 so that we neglected the aging feature of the viscous behavior. The ref-
174 erence time $t = 0$ corresponds to the time at which the load was applied
175 for the creep experiments. For concrete samples B28-1, B33-1A, B33-1B,
176 and B33-1D, the shrinkage experiments were terminated between 1289 and
177 1338 days after loading. For these samples, subsequent autogenous shrink-
178 age was estimated by extrapolating the experimental data with the function
179 $\epsilon_s = \epsilon_s^\infty (t/t_0)^a / ((t/t_0)^a - b)$, in which $t_0 = 1$ day and the parameters ϵ_s^∞ , a
180 and b were fitted for each sample.

181 *2.3. Months-long uniaxial compression creep experiments on cement paste*

182 On the cement paste samples, basic creep was measured, again by per-
183 forming in parallel an autogenous shrinkage test and a creep test on samples
184 with identical mix formulation and geometry. Autogenous shrinkage exper-
185 iments started 24 hours after mixing. Creep experiments under uniaxial
186 compression started 28 days after casting. The axial stress applied on sam-
187 ple P50-0SV was 9.4 MPa (i.e., about 28% of its compressive strength at 28
188 days after mixing). For all other cement paste samples, the applied stress
189 was 15.6 MPa (i.e., from about 13% to 22% of their compressive strengths at
190 28 days after mixing). The duration of the creep test was of about 100 days
191 for all samples. All tests were performed in sealed conditions at $20^\circ\text{C} \pm 2^\circ\text{C}$.

192 The basic creep strain $\epsilon_b(t)$ was again obtained as the difference between
193 the total axial strain $\epsilon_t(t)$ measured on the cement paste sample under load
194 and the axial strain $\epsilon_s(t)$ due to autogenous shrinkage and measured on the
195 cement paste sample subjected to no load: $\epsilon_b(t) = \epsilon_t(t) - \epsilon_s(t)$. From the

196 measured basic creep strain, the uniaxial creep functions $J_u(t)$ of the various
197 cement paste samples were obtained with Eq. (2). Here the reference time
198 $t = 0$ also corresponds to the time at which the load was applied for the
199 creep experiments, i.e., to 28 days after casting.

200 *2.4. Minutes-long microindentation creep experiments on cement paste*

201 On the cement paste samples, we also aimed at performing indentation
202 creep experiments, 28 days after casting. One day before testing, the samples
203 were moved into the room in which the microindenter was located and the
204 temperature was controlled at 23°C. About five minutes before testing, a
205 10-millimeters-thick disk was cut from the median part of the cylindrical
206 sample. The surface to be indented was then polished with 4 pads of silicon
207 carbide (SiC) paper with decreasing particle size. Polishing lasted for about
208 3 minutes, without any contact with water or other solvents. With respect
209 to the polishing procedure recommended by Miller et al. for nanoindentation
210 testing of cementitious materials [19], the duration of the procedure we used
211 here was much shorter. Indeed, since the scale of microindentation testing
212 is much larger than that of nanoindentation testing, our requirements on
213 surface roughness were much less strict than for those authors. In addition,
214 a rapid procedure also allowed to minimize drying. A typical surface with
215 a typical indent is presented in Fig. 1. As can be observed, the scale of
216 the indent is greater than the characteristic scale of the microstructure of
217 the cement paste: thus, the performed microindentation tests provided the
218 mechanical properties of the cement paste itself (and not of the individual
219 phases of which this cement paste is constituted).

220 The microindenter was calibrated according to the ASTM standard E

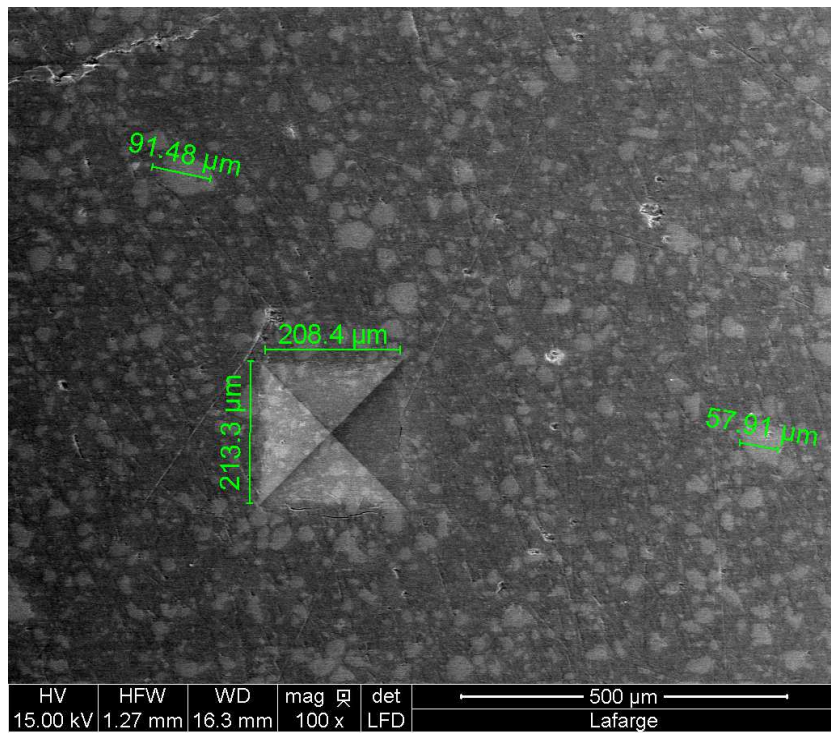


Figure 1: Scanning electron microscopy picture of the surface of an indented cement sample.

221 2546-07 [1]. On each sample, 10 microindentation tests were performed with
 222 a Vickers indenter probe and a maximal applied force of 20 N. For each
 223 indent, the load was increased linearly over time in 15 seconds, kept constant
 224 during the holding phase, and decreased linearly over time back to zero in
 225 15 seconds. Out of the 10 indents performed on each sample, 5 were with a
 226 relatively short 20-seconds-long holding phase, while 5 were performed with
 227 a relatively long 300-seconds-long holding phase. Indents with a 20-seconds-
 228 long holding phase enabled to measure the indentation modulus M_0 of the
 229 paste by following the ASTM standard E 2546-07 [1], which employs the
 230 Oliver and Pharr method [22]. The indentation modulus M_0 of the indented
 231 material is linked to its Young's modulus E_0 and Poisson's ratio ν_0 through
 232 [10]:

$$M_0 = \frac{E_0}{1 - \nu_0^2} \quad (3)$$

233 When reported in this work, the indentation modulus M_0 of a paste was
 234 obtained by averaging the 5 indentation moduli obtained from the 5 microin-
 235 dentation tests with a 20-seconds-long holding phase.

236 Indents with a 300-seconds-long holding phase enabled to measure the
 237 creep properties of the paste. For a linear viscoelastic material, upon in-
 238 dentation by a conical probe, the load $P(t)$ can theoretically be linked to
 239 the indentation depth $h(t)$ and its evolutions over time through the use of a
 240 so-called contact creep compliance $L(t)$ with [31]:

$$h^2(t) = \frac{\pi}{2 \tan(\theta)} \int_0^t L(t - \tau) \dot{P}(\tau) d\tau \quad (4)$$

241 where θ is the semi-apex angle of the conical probe. For the Vickers indenta-

242 tions performed in this study, the semi-apex angle of the equivalent conical
 243 probe is $\theta = 70.32^\circ$. The introduced contact creep compliance $L(t)$ has been
 244 shown to be a material property, i.e., a function that depends neither on the
 245 geometry of the probe, nor on the load used for the creep experiment [31].
 246 This contact creep compliance bears as much information on the viscoelastic
 247 properties of the material as the uniaxial creep compliance $J_u(t)$ introduced
 248 in Sec. 2.2. As was the case with the uniaxial creep compliance $J_u(t)$, the
 249 contact creep compliance $L(t)$ at time $t = 0$ is fully determined by the elastic
 250 properties [31]: $L(t = 0) = 1/M_0$.

251 However, upon conical indentation testing, Eq. (4) remains only theoret-
 252 ical. Because of the concentration of stresses at the tip of the indenter probe,
 253 the indented material is deformed plastically even at the lowest applied load.
 254 In spite of the occurrence of time-independent plasticity, the contact creep
 255 function $L(t) - L(0)$ can be back-calculated from the holding phase of a
 256 conical indentation creep experiment, as given by the following formula [31]:

$$L(t) - L(0) = L(t) - \frac{1}{M_0} = \frac{2a_u \Delta h(t)}{P_{max}} \quad (5)$$

257 where P_{max} is the applied load during the holding phase of the test, $\Delta h(t)$
 258 is the increment of the penetration depth of the indenter probe with respect
 259 to the indented surface during holding and a_u is the radius of the equivalent
 260 projected contact area between the indenter probe and the indented surface
 261 at the onset of unloading. The radius was estimated with the Oliver and
 262 Pharr method [22]. In the equation above, the reference time $t = 0$ corre-
 263 sponds to the instance when the load applied to the indenter tip reaches the
 264 maximum value, i.e., to the beginning of the holding phase. At this reference

265 time $t = 0$, the contact creep compliance $L(t)$ must be equal to $L(0) = 1/M_0$.
266 The indentation modulus M_0 could be measured from the unloading phase
267 of the microindentation test performed with the 300-seconds-long holding
268 phases: indeed, within the frame of linear viscoelasticity one can show that,
269 if the holding phase is sufficiently long and the unloading phase sufficiently
270 short, the indentation modulus measured with the Oliver and Pharr method
271 is unbiased by viscous effects and thus truly representative of the instantane-
272 ous elastic properties of the indented material [9, 32]. However, in our
273 work, we focus on the contact creep function $L(t) - L(0)$ rather than the
274 contact creep compliance $L(t)$ and Eq. (5) shows that the back-calculation
275 of such creep functions from microindentation creep experiments requires no
276 determination of the elastic indentation modulus M_0 .

277 On sample P38-0SV, one indentation creep experiment with a 1800-
278 seconds-long holding phase was also performed in order to determine the
279 shape of the creep function at longer term. For this last experiment, the
280 maximal load still was 20 N, the duration of the loading phase 15 s, and the
281 duration of the unloading phase 15 s.

282 The internal relative humidity of cement paste cured in sealed conditions
283 at temperature of 20°C varies from 90% to 98% at the age of 28 days [14, 25].
284 Therefore, in order to avoid drying during testing, all microindentation creep
285 experiments were performed in an environment with a relative humidity equal
286 to $91\% \pm 2\%$ and temperature equal to $23^\circ\text{C} \pm 0.2^\circ\text{C}$. By doing so, we expect
287 drying-induced strains to be negligible with respect to the strains induced by
288 creep. Moreover, at the age of 28 days, the autogenous shrinkage of cement
289 over the duration of the 300-seconds-long creep phase can be neglected with

290 respect to the strains induced by creep. As a consequence, the contact creep
291 compliance $L(t)$ is expected to characterize the basic creep of the cement
292 paste.

293 **3. Results**

294 *3.1. Raw results*

295 Figure 2 displays the uniaxial basic creep functions $J_u(t) - 1/E_0$ of the
296 cement paste samples obtained by uniaxial compression. In this figure, $t = 0$
297 stands for the time at which loading was applied. For samples with no
298 addition of silica fume, creep increased with the water-to-cement ratio w/c .
299 For samples with 10% of silica fume added, varying the water-to-cement
300 ratio w/c from 0.28 to 0.38 hardly varied the amplitude of creep. For a given
301 water-to-cement ratio (i.e., $w/c = 0.28$ or $w/c = 0.38$), adding 10% of silica
302 fume decreased creep. For given mix proportions, changing the clinker (i.e.,
303 using a clinker from Saint Vigor (sample P38-0SV) or from Saint-Pierre-la-
304 Cour (sample P38-0SL) slightly modified the creep of the paste: however,
305 after a few days, the difference between the two basic creep functions mostly
306 remained constant over time, which means that the difference between the
307 creep of those two pastes was mostly due to the time-dependent behavior of
308 the pastes during the first days of loading.

309 The basic creep functions of the concrete samples obtained by uniaxial
310 compression are displayed in Fig. 3. In a consistent manner with the results
311 obtained on cement pastes, this figure shows that basic creep of concrete
312 increased with the water-to-cement ratio w/c . A comparison of the results
313 for samples B33-1A, B33-1B, B33-1C, and B33-1D shows that, globally, creep

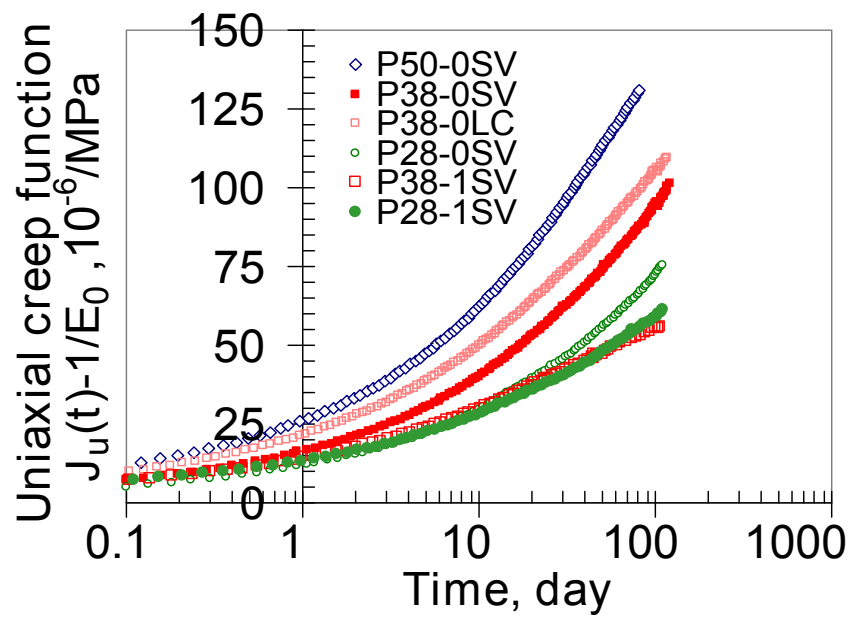


Figure 2: Uniaxial basic creep functions of cement paste samples obtained by uniaxial compressive creep testing. Experiments performed at LCPC (Paris, France), started by R. Le Roy and followed by F. Le Maou [16].

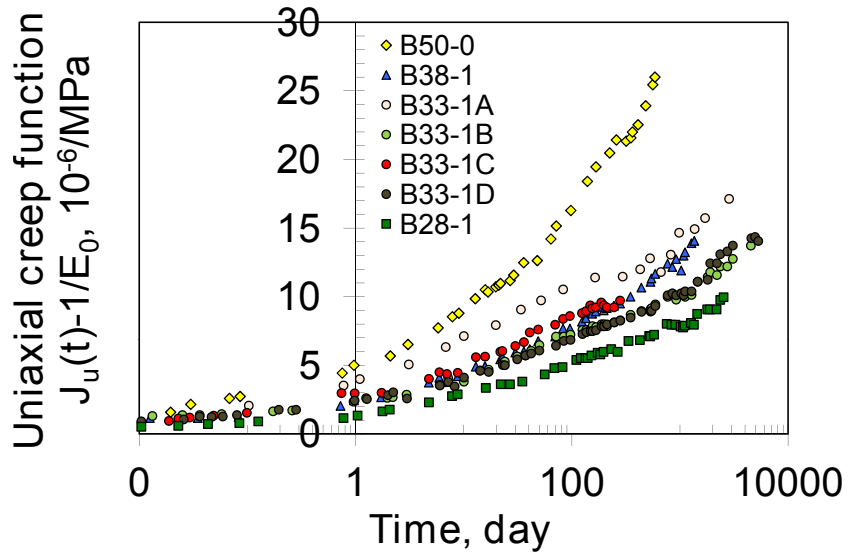


Figure 3: Uniaxial basic creep functions of concrete obtained from uniaxial compressive creep testing. Experiments performed at LCPC (Paris, France), started by R. Le Roy and followed by F. Le Maou [16].

314 decreases when the volume fraction of aggregates increases, though scattering
 315 hides this trend partially for sample B33-1B.

316 Microindentation creep experiments on cement pastes yielded contact
 317 creep functions $L(t) - 1/M_0$, which are displayed in Fig. 4. The follow-
 318 ing trends can be observed: the greater the water-to-cement ratio w/c was,
 319 the greater the magnitude of the creep strain was, and creep decreased with
 320 an addition of silica fume. Samples with different clinkers but with the same
 321 mix proportions (i.e., samples P38-0SV and P38-0LC) exhibited almost iden-
 322 tical creep. Note that the trends observed on the creep properties of cement
 323 pastes from microindentation test are qualitatively identical to the trends
 324 observed by macroscopic uniaxial compression test.

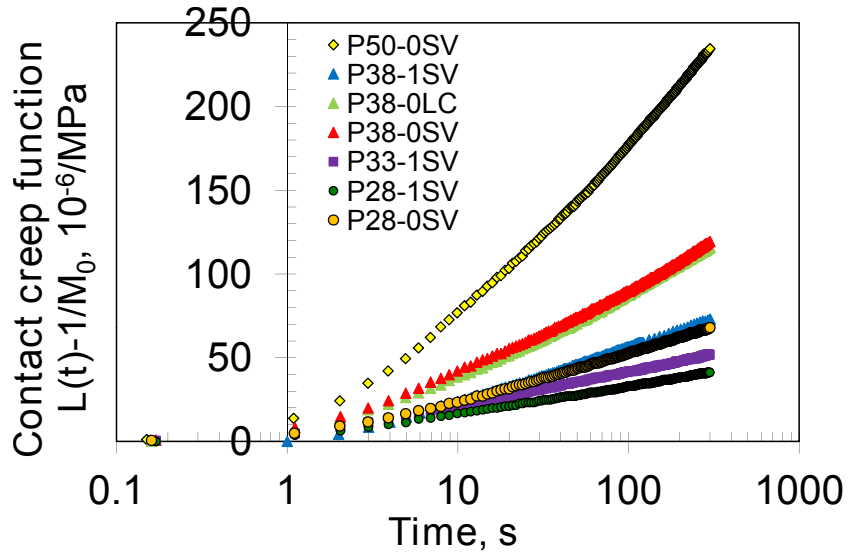


Figure 4: Contact creep functions $L(t) - 1/M_0$ of cement paste samples obtained by microindentation. For each sample, out of the 5 experiments performed, only the median curve is displayed.

325 *3.2. Direct comparison of microindentations on cement paste with uniaxial*
 326 *compressions on cement paste*

327 In this section, we aim at comparing results obtained by microindentation
 328 test with results obtained by regular macroscopic uniaxial testing, both in
 329 terms of elastic properties and in terms of creep properties.

330 For what concerns elastic properties, we explained in Sec. 2.4 how the
 331 indentation modulus M_0 of each sample was measured on each tested cement
 332 paste. Assuming a Poisson's ratio $\nu = 0.20$ for the samples, Eq. (3) enabled
 333 to calculate the Young's modulus E_0 of the indented cement pastes. The
 334 Young's moduli of the cement samples manufactured in 1992 were measured
 335 by Marchand by regular macroscopic compression [17]. Both sets of data
 336 are displayed in Fig. 5. The agreement between the Young's modulus of

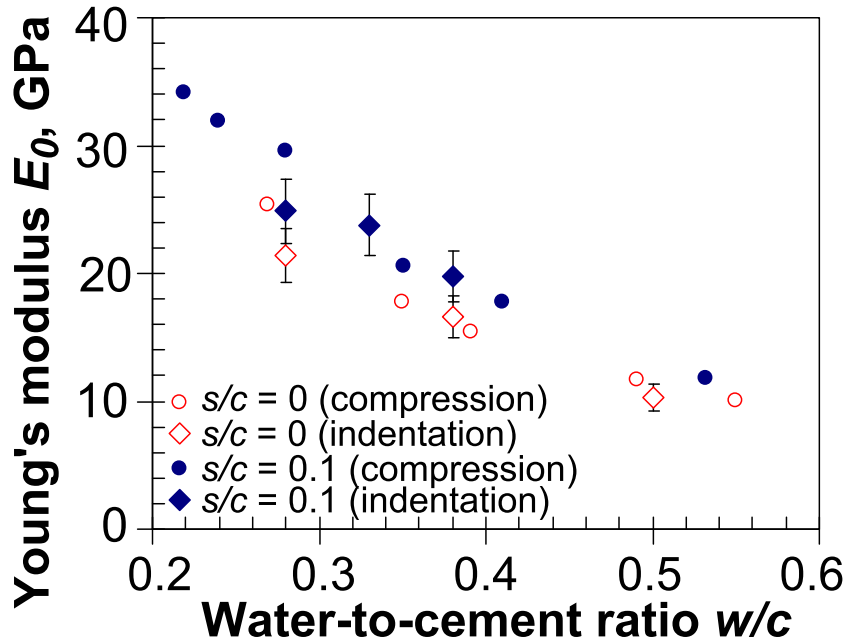


Figure 5: Young's modulus E_0 of the cement paste samples, determined by indentation test when assuming a Poisson's ratio $\nu = 0.20$ and determined by macroscopic uniaxial compression test. s/c stands for the mass ratio of silica fume to clinker. Experimental data for compression is from [17].

337 the cement pastes prepared in 2011 for indentation test and of the cement
 338 pastes prepared in 1992 for uniaxial testing is excellent, which proves that, by
 339 using similar raw materials and protocols of preparation, very similar cement
 340 pastes were prepared, although almost 20 years apart.

341 We now aim at comparing directly the creep functions obtained by uniax-
 342 ial test and by microindentation test. In order to do so, we focus on sample
 343 P38-0SV, on which a microindentation creep experiment with a 1800-seconds-
 344 long holding phase was performed. Both the derivative dL/dt with respect
 345 to time of the contact creep function obtained by microindentation test and

346 the derivative dJ_u/dt of the uniaxial creep function obtained by macroscopic
347 uniaxial test are displayed in Fig. 6. One readily observes that, over the
348 half hour compared, the rates measured by the two techniques differed by
349 one or even two orders of magnitude. In addition, a linear regression of this
350 plot in a log-log scale shows that, from 1 minute to 30 minutes, the rate of
351 the creep function measured by uniaxial test decreased as $t^{-0.51}$ while the
352 rate of the creep function measured by microindentation test decreased as
353 $t^{-0.99}$. From this simple comparison performed directly on creep function,
354 we conclude that the microindentation technique does not provide the same
355 creep function as macroscopic uniaxial test.

356 *3.3. Comparison of long-term logarithmic kinetics of creep*

357 *3.3.1. Principle*

358 From the microindentation creep data on cement pastes displayed in Fig.
359 4, one observes very clearly that, on the last two decades of the test, creep
360 was logarithmic with respect to time. For the microindentation performed
361 on sample P38-0SV with a 1800-seconds-long holding phase, creep was log-
362 arithmic on almost 3 decades (see Fig. 7c). Although less clearly, such
363 logarithmic kinetics of creep can also be observed on about one decade for
364 what concerns the uniaxial creep data on cement paste (see Fig. 2) and on
365 about one to two decades for the data on concrete (see Fig. 3). Moreover, this
366 kinetics is reminiscent of the long-term basic creep of cementitious materials,
367 which, as proposed by [several](#), can be well modeled by a logarithmic function
368 of time [2, 29]. Therefore, those observations suggest that not only years-
369 long uniaxial creep experiments on concrete and months-long uniaxial creep
370 experiments on cement paste, but —more surprisingly— also minutes-long

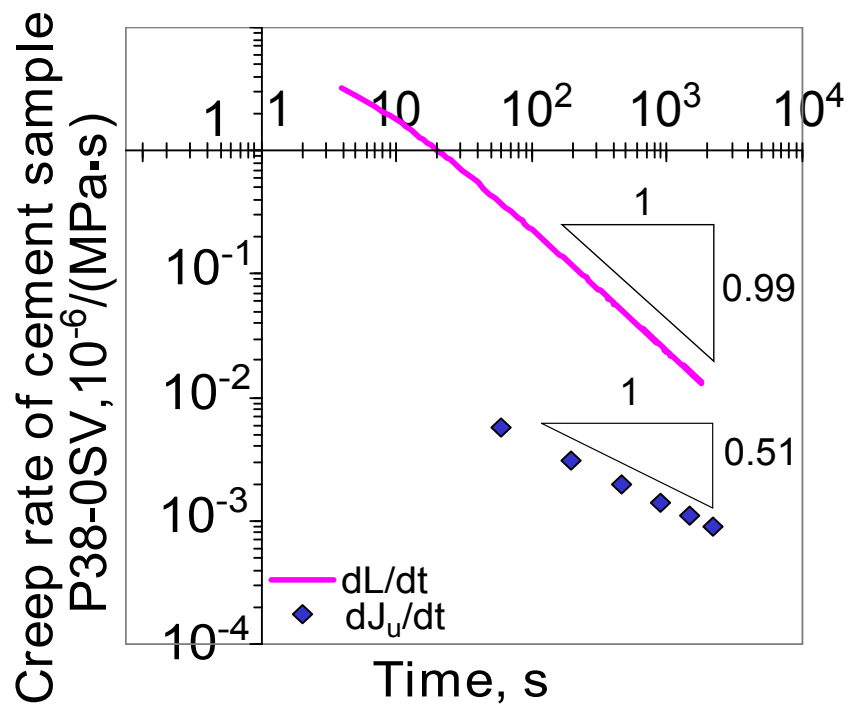


Figure 6: Derivatives with respect to time of the contact creep compliance obtained by microindentation test and of the uniaxial creep compliance obtained by macroscopic compression test on cement paste sample P38-0SV.

371 microindentation creep experiments on cement paste were all long enough in
 372 order to reach the long-term creep of the tested material, which exhibits a
 373 logarithmic kinetics.

374 In order to compare quantitative parameters, we fitted the measured creep
 375 data with logarithmic functions. More precisely, the indentation creep func-
 376 tion obtained on cement paste was fitted with:

$$L(t) - \frac{1}{M_0} = \frac{\ln(t/\tau_i + 1)}{C_i} \quad (6)$$

377 The uniaxial creep function obtained by uniaxial compression was fitted
 378 with:

$$J_u(t) - \frac{1}{E_0} = \frac{\ln(t/\tau_u + 1)}{C_u} \quad (7)$$

379 The parameters C_i and C_u are termed contact creep modulus and uniaxial
 380 creep modulus, respectively. The greater they are, the lower the amplitude
 381 of creep is. In the case of uniaxial testing, in order to differentiate between
 382 values obtained for cement paste and for concrete, the following notations
 383 are used: when the fit is performed for cement paste, the fitted parameters
 384 τ_u and C_u are noted $\tau_{u, cem}$ and $C_{u, cem}$, respectively; when the fit is performed
 385 for concrete, the fitted parameters τ_u and C_u are noted $\tau_{u, con}$ and $C_{u, con}$,
 386 respectively.

387 With such a choice of fitting functions, each creep function is condensed
 388 into 2 parameters: a characteristic time (noted τ_u when obtained from uni-
 389 axial testing and τ_i when obtained from indentation testing) and a creep
 390 modulus (noted C_u when obtained from uniaxial testing and C_i when ob-
 391 tained from indentation testing). The characteristic time characterizes the

392 time at which creep starts exhibiting a logarithmic kinetics. The creep mod-
 393 ulus governs the rate of this long-term kinetics:

$$\frac{dJ_u}{dt} \approx \frac{1}{C_u t} \text{ if } t \gg \tau_u \quad (8)$$

$$\frac{dL}{dt} \approx \frac{1}{C_i t} \text{ if } t \gg \tau_i \quad (9)$$

394 Figure 7 displays the best fits obtained with the functions introduced in
 395 Eqs. (6) and (7). The best-fit parameters are presented in Tables 5, 6, and
 396 7. Table 5 shows that the characteristic time needed to reach logarithmic
 397 kinetics varied tremendously with the type of sollicitation: this characteristic
 398 time was on the order of a day for uniaxial creep experiments on cement
 399 paste or on concrete, but was on the order of a second for the microinden-
 400 tation creep experiments on cement paste. Said otherwise, microindentation
 401 enabled to reach the logarithmic kinetics of creep orders of magnitude faster
 402 than regular macroscopic test.

403 3.3.2. *Comparison from paste to paste*

404 The contact creep modulus C_i fitted on the microindentation creep ex-
 405 periments on cement paste and the uniaxial creep modulus $C_{u, cem}$ fitted on
 406 the uniaxial creep experiments on cement paste are given in Table 6 and
 407 displayed in Fig. 8 with respect to each other. Fitting a linear relation to
 408 the experimental data through zero yielded $C_i = 1.198C_{u, cem}$ with an aver-
 409 age distance of the data points to the fitted line of 13.7 GPa. At least, the
 410 contact creep moduli and the uniaxial creep moduli were of the same order of
 411 magnitude, while the creep functions measured uniaxially and by indentation
 412 differed by more than one order of magnitude (see Fig. 8). However, given

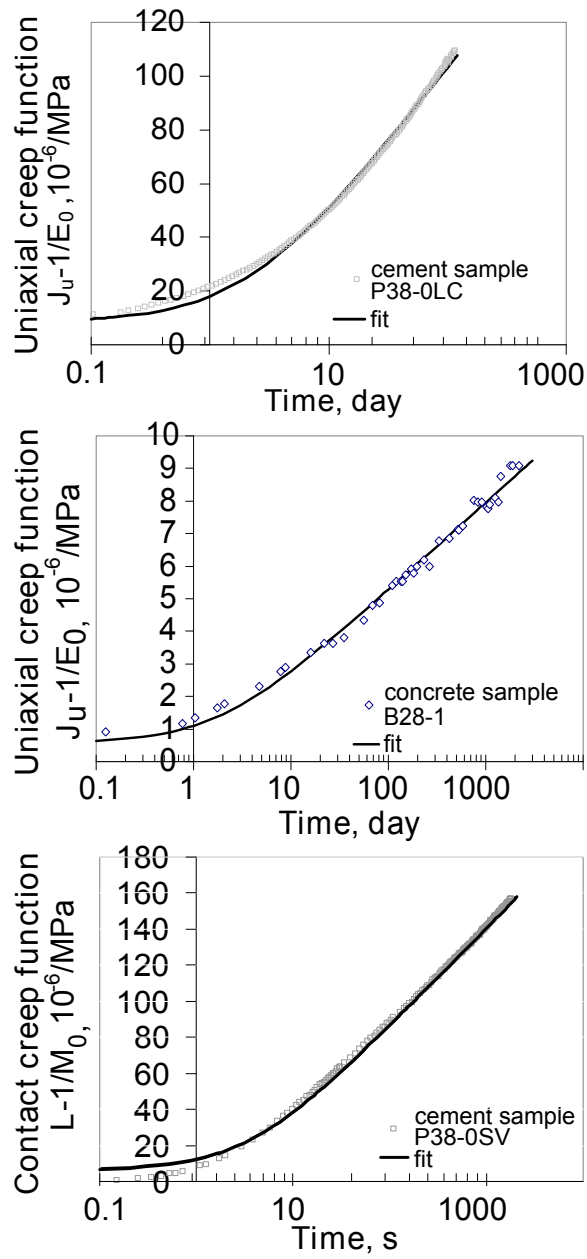


Figure 7: Examples of basic creep functions and of the best fits obtained with Eq. (6) or Eq. (7): a) uniaxial creep function obtained by uniaxial compression of cement paste P38-0LC, b) uniaxial creep function obtained by uniaxial compression of concrete B28-1, and c) contact creep function obtained by microindentation of cement paste P38-0SV.

Sample	$\tau_{u, cem}$ (day)	τ_i (second)
P28-0SV	4.8	2.2±0.6
P38-0SV	3.7	3.2±0.7
P50-0SV	2.6	2.4±0.8
P28-1SV	2.1	1.5±0.4
P33-1SV	-	1.3±0.4
P38-1SV	0.9	2.4±0.6
P38-0LC	2.1	3.3±0.3

Sample	$\tau_{u, con}$ (day)	
B28-1	2.2	-
B38-1	2.5	-
B50-0	2.3	-
B33-1A	0.2	-
B33-1B	1.4	-
B33-1C	0.5	-
B33-1D (data until 5320 days)	2.8	-
B33-1D (data until 1800 days)	1.1	-

Table 5: Characteristic time τ_u obtained by uniaxial compression creep experiment and τ_i obtained by indentation creep experiment.

Sample	$C_{u,cem}$ (GPa)	C_i (GPa)
P28-0SV	47.89	75.16
P38-0SV	39.42	39.68
P50-0SV	29.49	20.89
P28-1SV	75.16	124.5
P33-1SV	-	104.5
P38-1SV	94.10	67.46
P38-0LC	40.86	39.74

Table 6: Contact creep modulus C_i obtained by microindentation creep experiment on cement paste and uniaxial creep modulus $C_{u,cem}$ obtained by uniaxial compression creep experiment on cement paste.

413 the relative poorness of the fit displayed in Fig. 8, we conclude that the log-
414 arithmic creep measured by microindentation did not enable us to precisely
415 retrieve the amplitude of the logarithmic creep measured by macroscopic
416 uniaxial test on cement paste.

417 3.3.3. *Comparison from paste to concrete*

418 The contact creep moduli C_i were measured at the scale of cement paste,
419 while the uniaxial creep moduli $C_{u,con}$ were measured at the scale of concrete:
420 those two sets of moduli can therefore not be directly compared. In order
421 to make a comparison possible, results obtained at the scale of cement paste
422 must be upscaled to the scale of concrete. We performed this upscaling by
423 using homogenization techniques within the frame of linear viscoelasticity.
424 For details on how to perform homogenization of materials that creep loga-
425 rithmically with respect to time, we refer to the work of Vandamme and Ulm

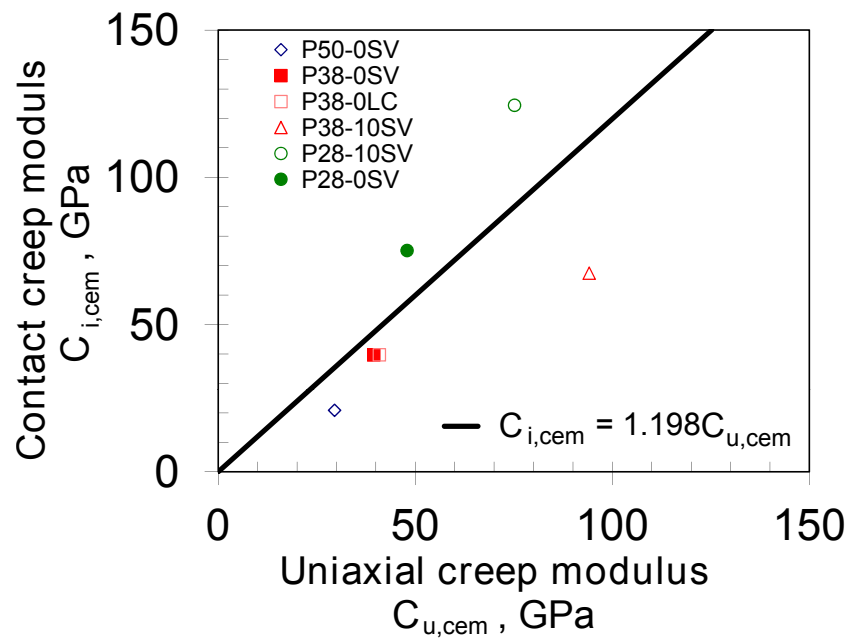


Figure 8: Uniaxial creep modulus $C_{u,cem}$ versus contact creep modulus C_i for cement paste samples.

426 [34]. In particular, these authors showed that, if a cement paste creeps loga-
 427 rithmically with respect to time in the long term, the concrete of which it is
 428 made (i.e., a mixture of creeping cement paste with non-creeping aggregates)
 429 should also creep logarithmically in the long term. Also, they showed that, if
 430 the cement paste creeps deviatorically in the long term **i.e., with no volume**
 431 **change**, a concrete made with this paste should also creep deviatorically in
 432 the long term. Making use of a Mori-Tanaka scheme, which is well adapted
 433 to matrix-inclusions morphologies, they showed that the contact creep mod-
 434 ulus $C_{i,con}$ of the concrete can be estimated from the contact creep modulus
 435 C_i of the cement paste with:

$$C_{i,con} = C_i \frac{2 + 3f_{agg}}{2(1 - f_{agg})} \quad (10)$$

436 where f_{agg} is the volume fraction occupied by the aggregates in the concrete.

437 Applying the above equation, for each concrete sample we estimated its
 438 contact creep modulus $C_{i,con}$ from the contact creep modulus C_i measured on
 439 the cement paste of which this concrete was made. Table 7 provides both this
 440 contact creep modulus $C_{i,con}$ estimated by microindentation tests performed
 441 at the scale of the paste and the uniaxial creep modulus $C_{u,con}$ measured by
 442 uniaxial compression of the concrete sample. Figure 9a displays those two
 443 creep moduli with respect to each other for all concrete samples. **Although**
 444 **this relation is roughly linear, we note that the data point associated to**
 445 **sample B33-1D falls outside this linear relationship.**

446 The uniaxial creep function of sample **B33-1D**, already displayed in Fig.
 447 3 together with the uniaxial creep functions of all other concrete samples
 448 tested, is displayed again in Fig. 10. On this latter figure, one can clearly

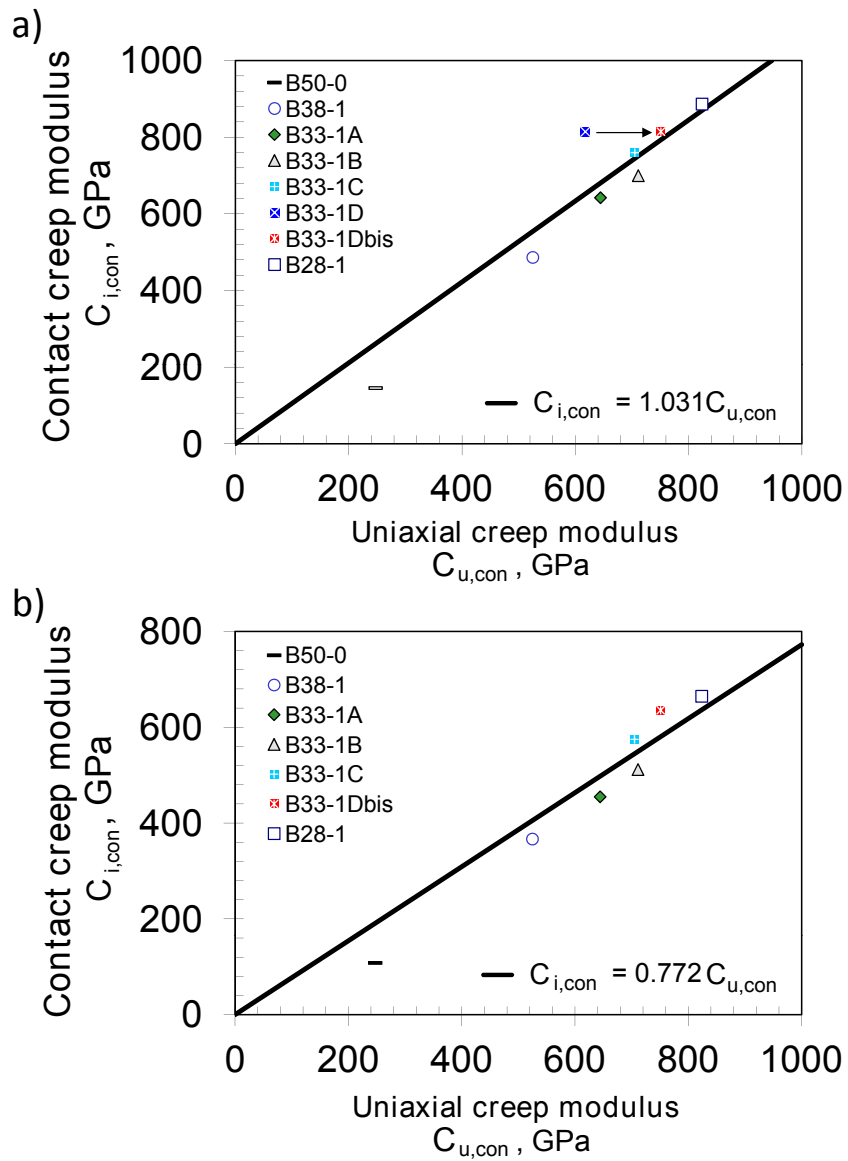


Figure 9: Uniaxial creep modulus $C_{u,con}$ measured by uniaxial compression creep experiments on concrete versus contact creep modulus $C_{i,con}$ of concrete estimated from microindentation creep experiments performed on cement paste, by considering, for the upscaling from the scale of cement paste to the scale of concrete, a) a Mori-Tanaka scheme and b) the upsizing model of Vu et al. [35].

Sample	$C_{u,con}$ (GPa)	$C_{i,con}$ (GPa)
B28-1	823.6	886.5
B38-1	524.8	486.4
B33-1A	644.3	642.2
B33-1B	711.1	699.8
B33-1C	705.0	759.9
B33-1D (data until 5320 days)	617.6	814.4
B33-1D (data until 1800 days)	751.0	
B50-0	247.3	145.7

Table 7: Uniaxial creep modulus $C_{u,con}$ measured by uniaxial compression creep experiments on concrete and contact creep modulus $C_{i,con}$ of concrete estimated from microindentation creep experiments performed on cement paste.

449 observe that the creep function of this concrete exhibited a nice logarithmic
450 dependency on time after about a dozen of days, but that the creep
451 rate sharply increased after about 1800 days. Wondering whether the data
452 gathered after 1800 days was still fully representative of the basic creep of
453 the sample, we performed again the analysis of the data on this sample, by
454 considering only data points up to 1800 days. A fit of the function given in
455 Eq. (7) to this new set yielded a new uniaxial creep modulus $C_{u,con} = 751.0$
456 GPa and a new characteristic time $\tau_{u,con} = 1.1$ day for this sample. The
457 $C_{i,con}$ -versus- $C_{u,con}$ relationship with this corrected uniaxial creep modulus is
458 also displayed in Fig. 9a.

459 One can observe that the correlation between uniaxial creep modulus
460 measured by macroscopic uniaxial test and contact creep modulus estimated

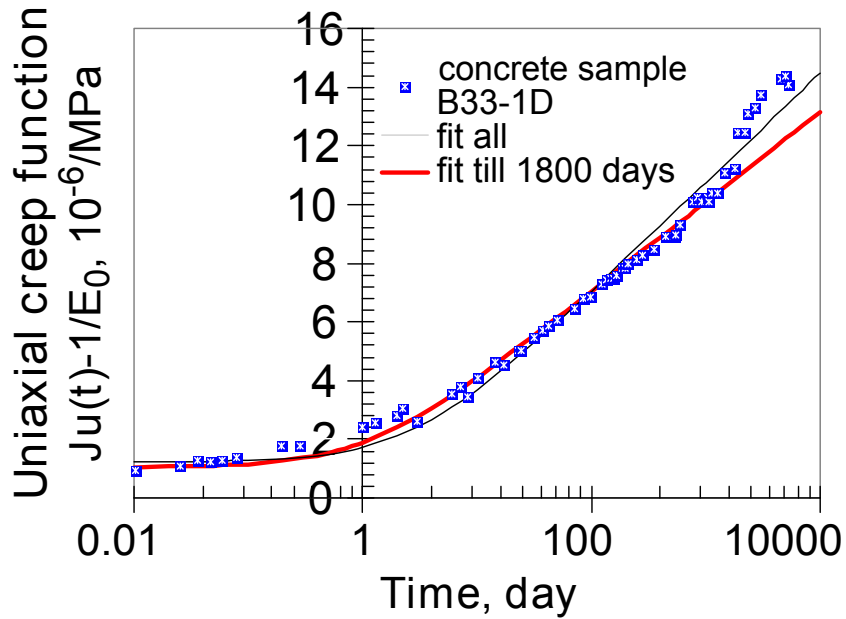


Figure 10: Basic creep function of concrete sample B33-1D, together with Eq. (7) fitted on all data points and with Eq. (7) fitted on data points until 1800 days only.

461 by microindentation at the scale of the cement paste is now much better. Fit-
 462 ting a linear relation to the **experimental data with the corrected data point**
 463 **for sample B33-1D** yielded $C_{i,con} = 1.031C_{u,con}$ with an average distance of
 464 the data points to the fitted line of 32.8 GPa: the agreement between di-
 465 rect measurements of long-term creep properties of concrete and estimations
 466 based on microindentation tests at the scale of the cement paste is excellent.
 467 Better than the comparison on cement paste, the creep moduli of various
 468 concrete samples estimated by microindentation at the scale of cement paste
 469 compared well with the creep moduli measured by macroscopic creep test, in
 470 spite of an extra step of homogenization.

471 **4. Discussion**

472 *4.1. On the coefficient between contact and uniaxial creep modulus*

473 For concrete, an excellent agreement was found between macroscopic mea-
474 surements and estimates based on microindentation tests (see Fig. 9a), and
475 the coefficient that enables to translate a contact creep modulus $C_{i,con}$ into a
476 uniaxial creep modulus $C_{u,con}$ was found **to be equal to** $C_{u,con} = 0.970C_{i,con}$.
477 **Why such a coefficient?** In elasticity the indentation modulus M_0 is linked
478 to the Young's modulus E_0 through Eq. (3). **Considering** that, on the
479 long term, the material of interest (here concrete) is linear viscoelastic and
480 creeps **only deviatorically (i.e., with no volume change)**, an application of
481 the s -multiplied Laplace transform to Eq. (3) yields **the theoretical relation**
482 $C_{u,con} = (1 - 0.5^2)C_{i,con} = 0.75C_{i,con}$. **Therefore, we observe a discrepancy be-**
483 **tween the theoretical coefficient and the one used experimentally to convert**
484 **the contact creep modulus into a uniaxial one. As will be seen in the next**
485 **section, this discrepancy may be explained by the choice of homogenization**
486 **scheme that we used to estimate the contact creep modulus of the concrete**
487 **from the one of the paste.**

488 *4.2. On the choice of homogenization scheme*

489 **In** order to predict the creep of concrete from the creep of the paste mea-
490 sured by indentation, we needed to employ a homogenization scheme, namely
491 the Mori-Tanaka scheme (see Eq. 10). At high volume fractions of inclusions,
492 the estimation given by the scheme is less accurate [11]. Therefore, one may
493 want to try other schemes. As an alternative, we employed the upscaling
494 model proposed by Vu et al. [35] for bidisperse suspensions of noncolloidal

495 particles in yield-stress fluids. For such suspensions, the shear modulus G
 496 of the suspension is related to the shear modulus G_0 of the suspending fluid
 497 through $G/G_0 = (1 - f/f_m)^{-1.43}$, where f is the volume fraction of the
 498 particles and f_m a critical volume fraction at which the elastic properties
 499 diverge. Adapting their model to our problem of a concrete made of non-
 500 creeping aggregates, we translated their formula for viscous properties as:
 501 $C_{i,con} = C_i(1 - f_{agg}/f_{agg,m})^{-1.43}$ or $C_{u,con} = 0.75C_i(1 - f_{agg}/f_{agg,m})^{-1.43}$, where
 502 $f_{agg,m}$ is the critical volume fraction of aggregates above which creep proper-
 503 ties should diverge. Fitting this relation to the experimental measurements
 504 yielded very satisfactory results for a critical volume fraction $f_{agg,m} = 90.4\%$
 505 of aggregates. For such parameter, a linear fit through zero of the relation
 506 $C_{u,con}$ -versus- $0.75C_{i,con}$ yielded $C_{u,con} = 0.978 \times 0.75C_{i,con}$ with an average
 507 distance of the data points to the fitted line of 48.4 GPa (see Fig. 9b).
 508 Therefore, we conclude that the discrepancy observed with the Mori-Tanaka
 509 scheme (i.e., the fact that the coefficient of proportionality observed between
 510 the measured uniaxial creep modulus of concrete and the contact creep mod-
 511 ulus of concrete predicted with the Mori-Tanaka scheme did not correspond
 512 to the value 0.75 expected theoretically) is likely due to the fact that the
 513 Mori-Tanaka scheme is not adapted to systems with high volume fractions of
 514 inclusions. At such high volume fractions, the upscaling model proposed by
 515 Vu et al. [35] may be more relevant. However, in turn, one should note that
 516 the use of the Mori-Tanaka scheme requires the knowledge of the mechanical
 517 properties of the individual phases and of their volume fractions only, while
 518 the use of the upscaling model of Vu et al. requires the additional knowledge
 519 of a critical volume fraction $f_{agg,m}$ of aggregates.

520 *4.3. On the ability of indentation to characterize long-term creep*

521 The comparison in the previous section shows that a 5-minutes-long mi-
522 croindentation test at the scale of the cement paste enables to quantitatively
523 predict the long-term logarithmic creep kinetics of a concrete sample. This
524 result, although already proposed by Vandamme and Ulm [33], is surpris-
525 ing, since this long-term logarithmic kinetics is only reached after days at
526 the scale of macroscopic samples, or even after years at the scale of struc-
527 tures [3]. According to Vandamme and Ulm, the ability to characterize long-
528 term creep kinetics so fast by microindentation is apparently not due to the
529 fact that microindentation probes a much smaller volume than macroscopic
530 experiments, or probe those volumes at much higher strains than regular
531 macroscopic test. They proposed the tentative explanation that this abil-
532 ity is due to the fact that microindentation probes the material at much
533 greater stresses than macroscopic test. In our study, for uniaxial creep ex-
534 periments, we observed no significant difference between the characteristic
535 time needed to reach a logarithmic kinetics of creep on cement paste or on
536 concrete (see Table 5). This observation further suggests that the charac-
537 teristic time needed to reach a logarithmic kinetics of creep is not governed
538 by the size of the system; on the other hand, the difference in the size of
539 the concrete samples (the length of which was 1000 mm and the diameter of
540 which was 160 mm) and of the cement paste samples (the length of which
541 was 160 mm and the diameter of which was 20 mm) may have been not
542 sufficient to observe significant differences between the characteristic times
543 for the two sets of samples. In addition, since this characteristic time did
544 not differ much between cement samples and concrete samples, we can also

545 conclude that the heterogeneity of the system does not modify the duration
546 needed to reach a long-term creep: indeed, concrete samples are more het-
547 erogeneous than cement paste samples, in the sense that concrete is itself a
548 mixture of cement paste with aggregates.

549 *4.4. On the quality of the creep experiments*

550 The quality of the correlation between microindentation results and macro-
551 scopic uniaxial results was much better at the scale of the concrete than at
552 the scale of the cement paste (see Figs. 8 and 9). Such a result is quite sur-
553 prising, since comparing results at the scale of a concrete sample required to
554 homogenize results obtained by microindentation at the scale of the cement
555 paste: by doing so, since homogenization schemes such as the Mori-Tanaka
556 scheme only provide estimates of the homogenized properties, one could have
557 expected that the quality of the correlation would have been worse at the
558 scale of the concrete than at the scale of the cement paste. Our opinion is
559 that the relatively poor correlation on cement paste samples is due to the
560 difficulty of performing creep experiments on cement samples. In general,
561 performing creep experiments on cementitious materials is tricky and, even
562 when great care is taken, a dispersion of the **long-term** creep results of about
563 **16.5%** can be expected on concrete samples **tested 28 days after casting** [5].
564 **For younger samples, this dispersion is rather on the order of 20%** [5]. On
565 cement paste samples, even more dispersion should be expected, since prepar-
566 ing the samples proves to be very delicate, in spite of the fact that cement
567 paste samples are smaller than concrete samples. An example of such a dif-
568 ference in the difficulty of preparing both sets of specimen is the fact that
569 cement paste samples needed to be rotated for a few hours after casting in

570 order to prevent segregation, while concrete samples did not.

571 In order to obtain Fig. 9b from Fig. 9a, we needed to perform a new
572 analysis of the creep data of sample B33-1D. For this sample, instead of
573 considering all the data available on the 5320 days (i.e., about 14.5 years),
574 we only considered data on about 1800 days (i.e., about 5 years), because of
575 a kink in the data at about 1800 days, that we considered as spurious (see
576 Fig. 10). Although the spuriousness of this kink can be discussed, we want
577 to underline how difficult and tedious running creep experiments on such
578 long periods is. Since the objective of such experiments is to measure basic
579 creep, on several years the temperature must be well controlled and all hygric
580 exchanges must be prevented. The difficulty is also enhanced by the fact that
581 basic creep strain is measured by difference between the strains measured
582 on a loaded sample and on an unloaded sample (see Sec. 2.2): therefore,
583 basic creep will be correctly measured only if temperature is well controlled
584 and hygric exchanges are prevented for both samples, thus increasing the
585 risks of experimental error. In our present study, experimental error due
586 to temperature variations must have been negligible, since all samples for
587 compressive creep experiments and autogenous shrinkage experiments were
588 located in the same room and thus at the same temperature. In contrast,
589 since upon years drying can occur even for samples tightly sealed with self-
590 sealing aluminum foil [28], experimental error due to long-term drying can
591 not be discarded, in particular for sample B33-D.

592 As an alternative to tedious years-long macroscopic experiments, minutes-
593 long microindentation testing would prove to be very handy. And our work
594 showed that such microindentation testing makes it possible to characterize

595 precisely the long-term logarithmic kinetics of creep of cementitious materi-
596 als. As a counterpart, this result means that microindentation testing cannot
597 give access to the short-term kinetics of those materials. Consequently, mi-
598 croindentation testing should be used as a complement to shorter macroscopic
599 creep experiments: the macroscopic experiments would enable to characterize
600 the short-term creep of the material and should be sufficiently long to reach
601 the long-term logarithmic kinetics of creep; while microindentation tests run
602 in parallel would enable to characterize the rate of this logarithmic kinetics
603 of creep. For practical use, other sources of creep (e.g., drying creep) would
604 need to be added to the long-term basic creep determined in such a manner.

605 **5. Conclusions**

606 This work was dedicated to comparing microindentation creep experi-
607 ments on cement paste with macroscopic uniaxial creep experiments on both
608 cement paste and concrete. Samples for uniaxial experiments were manufac-
609 tured in 1992, while samples for indentation test were manufactured in 2011.
610 Although the two sets of samples were prepared almost 20 years apart, we
611 used virtually the same raw materials and employed the same procedures of
612 preparation, so that the mechanical properties of both sets of cement pastes
613 could be expected to be very close to each other (see Fig. 5).

614 Uniaxial creep experiments lasted for years on concrete samples and for
615 months on cement paste samples. In contrast, microindentation creep ex-
616 periments (performed at the scale of cement paste) only lasted for minutes.
617 The creep rate measured by microindentation differed by one to two orders
618 of magnitude from the creep rates measured during the first thirty minutes

619 of the macroscopic uniaxial experiments (see Fig. 6): microindentation did
620 not provide access to the short-term creep of the tested cement pastes.

621 For all experiments, after a transient period, the basic creep was well
622 captured by a logarithmic function of time. The amplitude of the rate of this
623 logarithmic kinetics of creep depends on a creep modulus, called uniaxial
624 creep modulus for uniaxial creep experiments and contact creep modulus for
625 microindentation creep experiments. We compared the contact creep moduli
626 with the uniaxial creep moduli. The comparison with macroscopic uniax-
627 ial experiments on concrete required to homogenize the microindentation
628 results: this homogenization was performed within the frame of linear vis-
629 coelasticity. Contact creep moduli of concrete were in an excellent agreement
630 with uniaxial creep moduli measured by regular macroscopic test (see Fig.
631 9b). This result shows that the rate of long-term creep of concrete can be
632 quantitatively inferred from minutes-long microindentation experiments at
633 the scale of the cement paste. However, the coefficient of proportionality ob-
634 served between measured uniaxial creep moduli and predicted contact creep
635 moduli did not correspond to the value expected theoretically: this discrep-
636 ancy was attributed to the inaccuracy of the Mori-Tanaka scheme for systems
637 with high volume fraction of inclusions. At such high volume fractions, the
638 upscaling model proposed by Vu et al. [35] may be more relevant.

639 The measured contact creep moduli compared worse with the uniaxial
640 creep moduli on cement paste (see Fig. 8). We attributed this less good
641 agreement to the difficulty of measuring basic creep of cement pastes by
642 regular macroscopic testing. Several factors can make this measurement
643 tricky: difficulty of preparing homogeneous samples (which need to be rotated

644 after mixing), need to perform two experiments in parallel (since basic creep
645 is obtained by subtracting autogenous shrinkage to total creep), difficulty of
646 preventing hydric exchanges with the surroundings over long periods of time
647 and scattering due to a smaller volume compared with concrete.

648 The characteristic time needed to reach a logarithmic kinetics of creep
649 was of a few days with macroscopic uniaxial testing and of a few seconds
650 with microindentation testing. This striking observation – that small-scale
651 experiments enable to reach long-term creep of cementitious materials orders
652 of magnitude faster than macroscopic experiments – was already observed at
653 the scale of nanoindentation testing [34]. This surprising feature is appar-
654 ently not due to the fact that microindentation or nanoindentation probes
655 small volumes, or probes those volumes at large strains, or probes volumes
656 that are less heterogeneous than macroscopic ones. In contrast, a tentative
657 explanation proposed by Vandamme and Ulm [34] is that indentation testing
658 probes volumes at very large stresses, thus allowing for a fast redistribution
659 of internal stresses within the solid.

660 Our study shows that microindentation experiments provide access to
661 the long-term kinetics of creep of cementitious materials in minutes. As a
662 counterpart, such microindentation experiments do not allow to characterize
663 the short-term creep of those materials. From an engineering perspective,
664 microindentation could prove very beneficial, when used in parallel with reg-
665 ular macroscopic testing: the latter should only last long enough in order
666 to measure the short-term kinetics of creep, while the rate of the long-term
667 logarithmic creep would be characterized by microindentation. By doing so,
668 the whole basic creep function of cementitious materials could be measured

669 precisely and in a more convenient and faster way than is done today.

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