

Experimental study on the combined effect of temperature and mix parameters on the premature cracking of cement-based materials

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ABSTRACT: Cracking can occur in early-age concrete when thermal, hydrous or physico-chemical deformations are restrained. In addition to a deterioration of the aesthetic aspect, this premature cracking generates a loss of durability of the structure. In this article, the autogenous cracking of cement-based materials is studied using the ring test method. A specific experimental system based on this method was developed. It enables to keep the specimens in autogenous and temperature-controlled conditions during the whole test duration. The influence of both temperatures between 20°C and 40°C and the degree of restraint on the first autogenous cracking age of CEM I cement pastes with water-to-cement ratio $W/C = 0.3$ and 0.4 has been examined. Increasing the curing temperature and the degree of restraint and decreasing the W/C ratio leads to a shortening of the cracking age. Moreover, the level of maturity (hydration degree) corresponding to the age of the first autogenous cracking decreases when the curing temperature increases.

1 INTRODUCTION

At an early age, the volume changes of cement-based materials are the combined result of the physico-chemical evolution, the hydration heat release and the drying of the material. An inaccurate estimation of the amplitude and the rate of these volume variations can constitute an important cause of concrete durability problems.

In low water-to-cement ratio concretes ($W/C < 0.5$), the high cement proportioning results in an increase in the mechanical strength but also induces an augmentation of both autogenous and thermal deformations, in particular at a very early age. These deformations generate, when restrained, microscopic cracking (around the aggregates) or crossing cracks.

The knowledge of the deformation evolution in free conditions is not sufficient to precisely predict the risk of early-age cracking. Indeed, in restrained conditions, the relaxation phenomenon of the hydrating matrix attenuates the amplitude of autogenous shrinkage: the strain evolution is then different from that recorded in free conditions. It is therefore necessary to develop specific devices enabling the quantification of deformation in restrained conditions, taking into account the thermal history of the material. The parameters conditioning the development of early-age deformations are numerous: the cement type, the concrete's composition, the geometry and dimensions of the specimen, the curing temperature, the drying conditions, etc. The effects of

some of these parameters have been investigated in previous studies (Grzybowski et al. 1990, Weiss et al. 1999, Weiss & Ferguson 2001, Hossain & Weiss 2006), but early-age autogenous cracking and the temperature effect on this type of cracking did not receive much attention.

Several testing techniques exist for the determination of restrained shrinkage and cracking risk of cement-based materials (Bentur & Kovler 2003). Among them, an easy and simple method is the ring test. It consists in casting a specimen of cement-based material around a metal ring and in measuring the dimensional variations of this ring. The restrained shrinkage causes the development of tensile stresses in the material. As soon as they become higher than the material tensile strength, a transversal crack appears in the specimen.

The ring test method has been used in many research works (Table 1) for the quantification of the cracking risk of cement-based materials. A bibliography and an analysis of these studies have been recently presented by Radlinska et al. (2006).

The present work focuses on the early-age autogenous cracking in cement paste. The effects of three parameters are measured: the isothermal curing temperature, the degree of restraint and the initial amount of water (W/C ratio). The ring test device used in this study is equipped with a peripheral hermetic thermal regulation system, which enables to ensure quasi-isothermal and autogenous conditions during the test.

Table 1. Recent studies using the ring test method.

References	Mixture parameters			Test conditions	
	C	W/C	A/C	T (°C)	RH (%)
Grzybowski & Shah 1990	1	0.5	2	20	40
Weiss & Ferguson 2001	1	0.5	1.5	21	50
Weiss & Shah 2002	NSC	0.5	2.8	30	40
Hossain & Weiss 2006	1	0.3-0.4	1.5-1.4	23	50
Shah & Weiss 2006	1	0.5	1.8	23	50

C: cement type; NSC: normal strength concrete; W/C: water-to-cement ratio; A/C: aggregate-to-cement ratio; T: curing temperature; RH: relative humidity

2 EXPERIMENTAL PROGRAM

2.1 Materials

French CEM I cement is used for all the experiments. According to Bogue's formula, it is composed of 60.4% of C_3S , 12.2% of C_2S , 8.8% of C_3A , 8.0% of C_4AF and 7.4% of gypsum; its specific surface is $3390 \text{ cm}^2/\text{g}$.

Two water-to-cement ratios ($W/C = 0.3$ and 0.4) and three curing temperatures ($T = 20^\circ\text{C}$, 30°C and 40°C) are studied.

2.2 Quasi-isothermal ring-test device

The ring test device is schematized in Figures 1a and 1b. Figure 2 presents an overall photograph of the experimental system.

Each device is composed of two concentric rings: a PVC external ring and a metallic central ring. Three different metals (brass, steel and stainless steel) are used. The characteristics of these central rings are given in Table 2. Each cement paste specimen cast between the two rings has a cross section of $40 \times 40 \text{ mm}$.

The strain of the central annulus due to the cement paste shrinkage is measured by four strain gauges (Fig. 1b). Four additional gauges are placed inside the central ring for thermal compensation.

In order to carry out the tests at a controlled temperature, each device is equipped with a peripheral thermal regulation system made of a copper tube network joined on two aluminium plates and fed by a thermostated water bath ($\pm 0.1^\circ\text{C}$) via a pump (Fig. 2). To avoid thermal losses, insulating foam is placed on the copper tubes and each device is set in a wooden box (Fig. 1a).

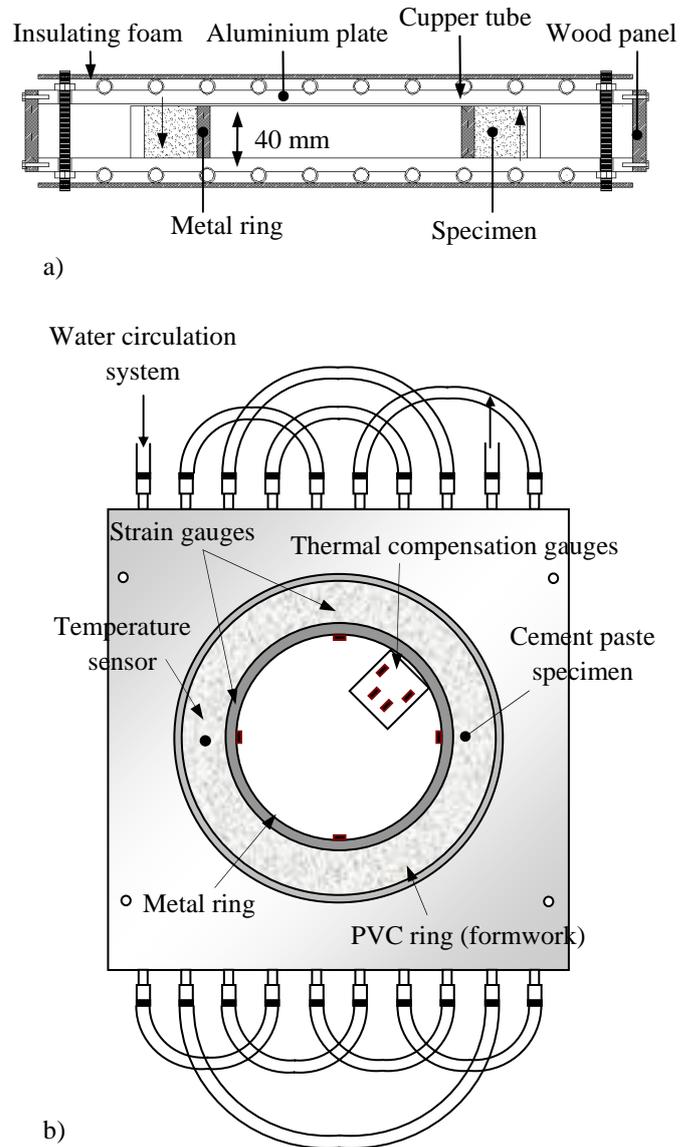


Figure 1. Diagrams of the quasi-isothermal ring-test device: a- side view and b- top view.

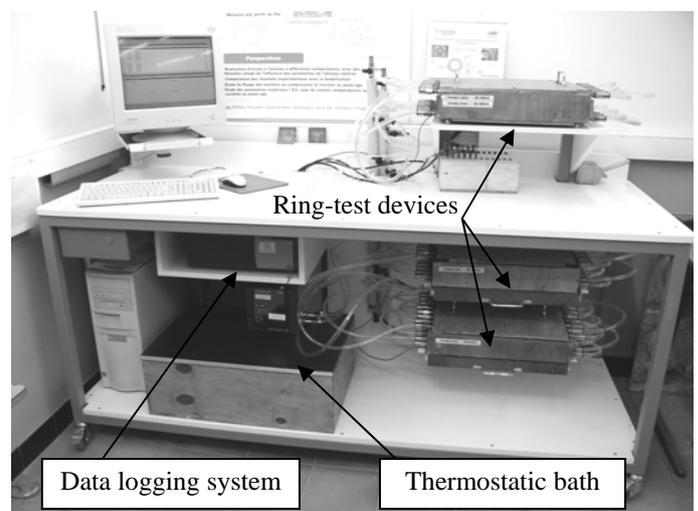


Figure 2. Photography of the experimental system.

Table 2. Characteristics of the metal rings.

Designation	R _{IS} mm	R _{OS} mm	E _m MPa	ν
Brass	95	105	100 000	0.33
Steel	95	105	211 000	0.30
Stainless Steel	95	105	200 000	0.30

R_{IS}: inner metal radius; R_{OS}: outer metal radius; E_m: elastic modulus; ν: Poisson coefficient.

2.3 Testing protocol

The water circulation in the thermal regulation system is activated two hours before the casting of cement paste in order to stabilize the temperature of the rings. Form oil is applied on the rings and on the bottom of the device to limit friction between the specimen and the ring walls.

Each cement paste is prepared by mixing cement with water for 3 minutes. The specimen is then immediately cast around the central metal ring. A thermocouple, embedded in the sample, makes it possible to follow the temperature of the material during the test. The wooden box is then sealed and the automatic measurement acquisition begins. The time lag between the end of mixing and the first strain measurement does not exceed 15 minutes. The deformation of the central ring and the temperature of the specimen are recorded by a data logging system every 20 minutes. The test is stopped at the appearance of the first crossing crack (Fig. 3) detected by a brutal jump on the strain curve of the metal ring.

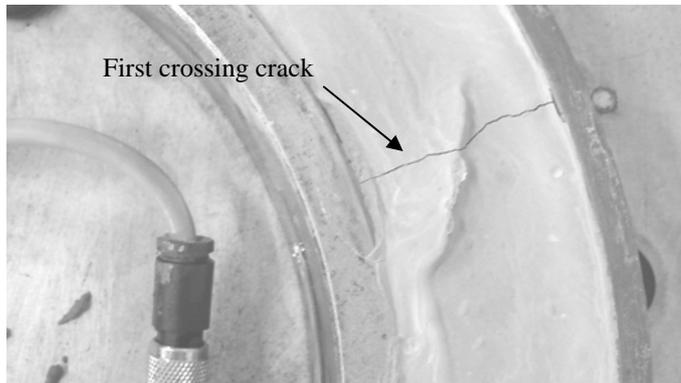


Figure 3. Transversal cracking in the cement paste annulus.

3 TEST RESULTS

3.1 Validation tests

3.1.1 Temperature control

The hydration heat can cause a significant elevation of the cement paste temperature during the first hours after mixing. It is essential to control these temperature variations in order to eliminate potential problems of thermal deformations.

Figure 4 shows the typical temperature curve of specimens during the tests. The beginning of each

curve is marked by a temperature variation of about $\pm 2^\circ\text{C}$ explained by the strong hydration heat release and the thermal inertia of the regulation system. This thermal instability lasts approximately 6 hours. Beyond this period, a maximal thermal deviation of 0.5°C is observed. The efficiency of the thermal regulation system can thus be considered as correct.

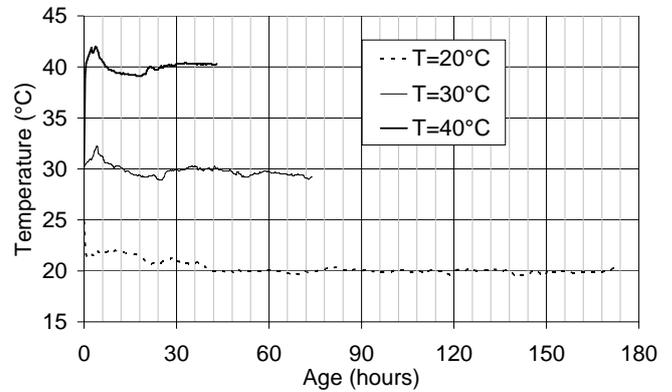


Figure 4. Temperature control of the cement paste specimens.

3.1.2 Repeatability tests

Figure 5 provides the result of three repeatability tests carried out with the brass ring and the W/C = 0.3-cement paste. The curing temperature is 40°C .

The repeatability level of the test method is acceptable. The strain curves follow the same evolution. The absolute difference in maximal strain amplitude and in cracking age is about $2 \mu\text{m/m}$ and 2.3 hours, respectively.

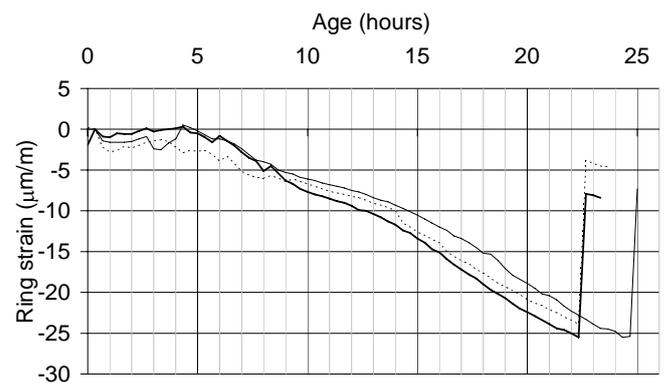


Figure 5. Repeatability of the ring tests (Brass ring, W/C = 0.3, T = 40°C).

3.2 Parametric study

In the three next paragraphs, the effects of the ring nature, the water-to-cement ratio and the temperature on the appearance of the first crack will be analysed.

3.2.1 Effect of the degree of restraint

Recently, Hossain & Weiss (2006) studied the effect of the degree of restraint by measuring the influence of the ring thickness on the age of the first crack in drying conditions. They noted that the increase of the degree of restraint causes a shortening of the cracking age.

In the present work, the effect of the degree of restraint is investigated by using metal rings with different elastic moduli (brass, stainless steel and steel). The degree of restraint R is computed using the expression proposed by See et al. (2003):

$$R = \frac{A_m \cdot E_m}{A_m \cdot E_m + A_c \cdot E_c} \quad (1)$$

where A_c and A_m are the section areas of the cement paste specimen ($A_c = 16 \text{ cm}^2$) and the metal ring ($A_m = 4 \text{ cm}^2$), respectively. E_c and E_m are the elastic moduli of the cement paste and the metal, respectively.

In the case of cement paste with $W/C = 0.3$, the values of the degree of restraint computed are 64% for the steel ring, 63% for the stainless steel ring and 46% for the brass ring. The effect of this degree of restraint can be seen on figure 6, which provides the results of the ring tests performed with the cement paste specimens prepared with $W/C = 0.3$ and cured at 30°C . From the ring strain ε_m , it is possible to calculate the residual stress σ_{residual} at the interface between the metal ring and the cement paste as (Weiss & Shah 2002, Hossain & Weiss 2004):

$$\sigma_{\text{residual}}(t) = -\varepsilon_m(t) \cdot E_m \cdot C_R \quad (2)$$

where C_R is the ring geometry coefficient:

$$C_R = \frac{R_{OS}^2 + R_{OC}^2}{R_{OC}^2 - R_{OS}^2} \times \frac{R_{OS}^2 - R_{IS}^2}{2R_{OS}^2} \quad (3)$$

with R_{OS} = outer metal radius; R_{OC} = outer cement paste ring radius; and R_{IS} = inner metal radius.

Figure 7 shows the evolution of the residual stress as a function of time for the three metal rings.

The three curves plotted in Figure 6 (and in Fig. 7) experience the same type of evolution: firstly a period of about 24 hours with low ring deformations (residual stress), then a faster augmentation of the strains (of the residual stress) and finally a brutal jump (drop) corresponding to the cracking of the specimens.

Figures 6 and 7 demonstrate that the nature of the central ring influences both the age of cracking and the maximal ring strain measured. The test performed with the brass ring, which has the lower rigidity, shows the later the age of cracking, the higher the ring strain and the residual stress: a reduction of 13% of the degree of restraint leads to double the amplitude of the ring strain and to increase the maximal residual stress of about 15%.

A difference of about 10h is recorded between the age of cracking obtained with the steel ring and that measured with the stainless steel. This difference cannot be explained only by the slight difference existing between the elastic moduli of these two rings. The surface quality of the rings, which induces more or less friction at the interface with the cement paste specimen, could also play an important role in this cracking age difference. The metal surface in contact with the specimens was observed with a video-microscope for each ring (Fig. 8). Different scratch thicknesses have been observed for the three rings. It can thus be supposed that the intensity of friction forces will not be the same for the three types of rings. However at the current state of our knowledge, it is still difficult to quantify the effect of the surface quality on the difference in cracking age. Tests are in progress to determine precisely the depth of roughness of each ring.

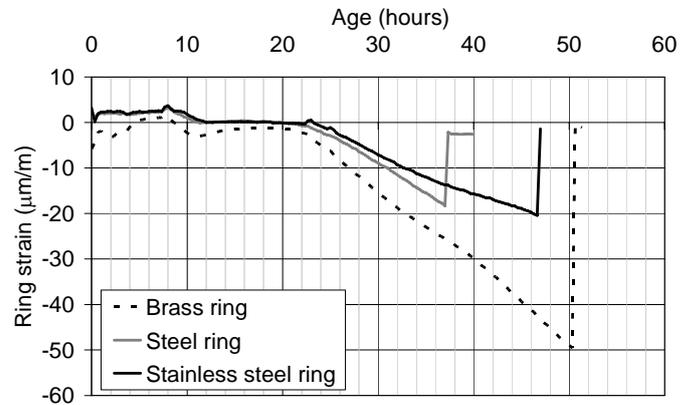


Figure 6. Ring strain for cement paste with $W/C = 0.3$ at $T = 30^\circ\text{C}$.

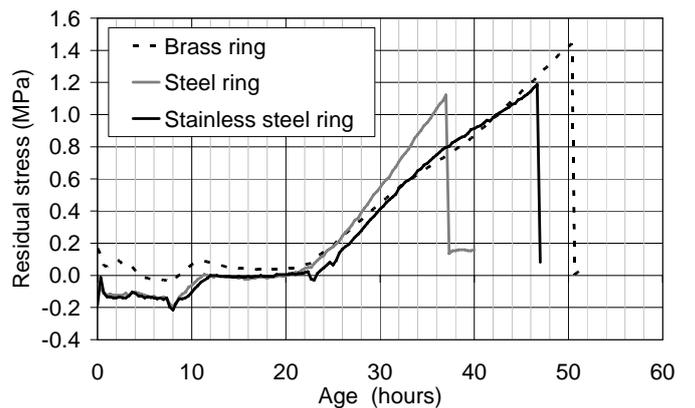


Figure 7. Residual stress for cement paste with $W/C = 0.3$ at $T = 30^\circ\text{C}$.



Figure 8. Surface quality of the rings (enlargement = $\times 50$).

3.2.2 Curing temperature effect

The second parameter studied is the curing temperature. Figure 9 gives the ring test results obtained for the specimens with $W/C = 0.4$ at 20, 30 and 40°C.

A preliminary point, which has to be underlined, is that, in the range considered, the temperature did not significantly modify the elastic modulus of metals: for instance, for stainless steel, it is 200 GPa at 20°C and 199 GPa at 40°C. In the following, the influence of this variation will be thus neglected.

Figure 9 shows that a higher curing temperature causes an acceleration of the early-age cracking: from 20 to 40°C, the cracking age is divided by 8.8. This phenomenon can be attributed to the thermo-activation of the cement hydration process at a very early age: when the temperature increases, the deformations of the cement paste develop faster (Turcry et al. 2002, Mounanga et al. 2006) and the value of cracking tensile stress is reached more quickly.

In fact, this acceleration is the result of the competition between several complex phenomena: on one hand, the increase of internal tensile stress due to autogenous shrinkage (partially counterbalanced by the creep relaxation of the hydrating matrix) and, on the other hand, the development of the material tensile strength. When the internal stress becomes higher than the tensile strength, the specimen cracks (Fig. 10). Temperature accelerates both the internal stress rate and the tensile strength evolution. According to our results, the fact that early-age cracking occurs sooner when the curing temperature is higher seems to demonstrate that the thermo-activation of the internal stress rate is more important than the thermo-activation of the tensile strength evolution.

Besides, a second effect of the increase of curing temperature is the diminution of the maximal ring strain measured just before the cracking of the specimen. Between 20°C and 30°C and for all the specimens investigated, a significant diminution of the maximal ring strain is recorded when temperature increases whereas the difference between the amplitudes of the maximal ring strain obtained at 30°C and at 40°C is much lower (Fig. 9).

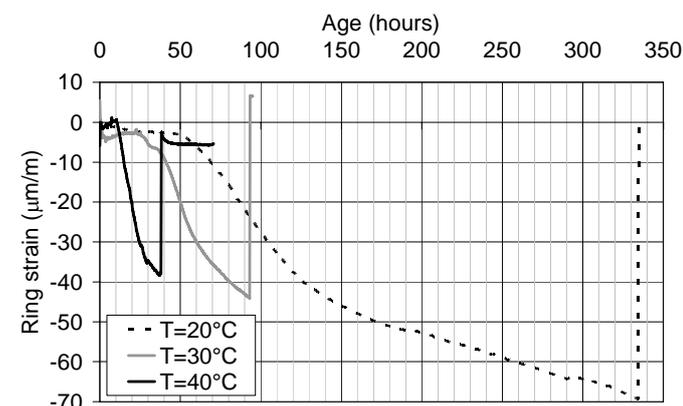


Figure 9. Brass ring strain for cement paste with $W/C = 0.4$ at $T = 20, 30$ and 40°C .

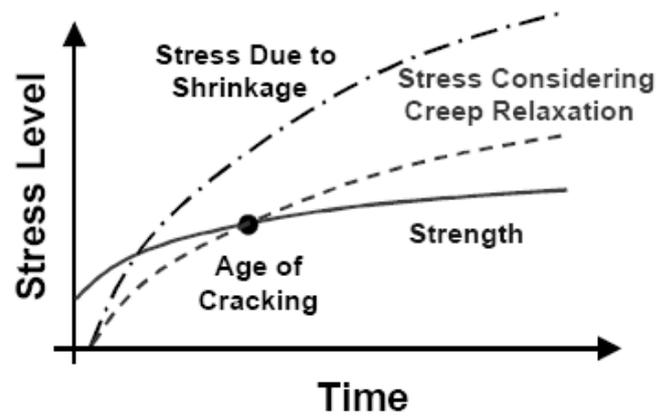


Figure 10. Competition between internal stress evolution and strength development in hydrating cementitious systems (Weiss 1999).

3.2.3 W/C ratio effect

Figures 11 and 12 show the effect of the W/C ratio on the brass ring test results at 20°C and 40°C, respectively. The initial amount of water in the material influences both the rate of the ring strain and the age of cracking. A difference of cracking age of about 215 hours is observed between the two cement pastes ($W/C = 0.3$ and $W/C = 0.4$) at 20°C. This difference becomes lower at 40°C, because of the thermo-activation of early age cracking discussed in the previous section.

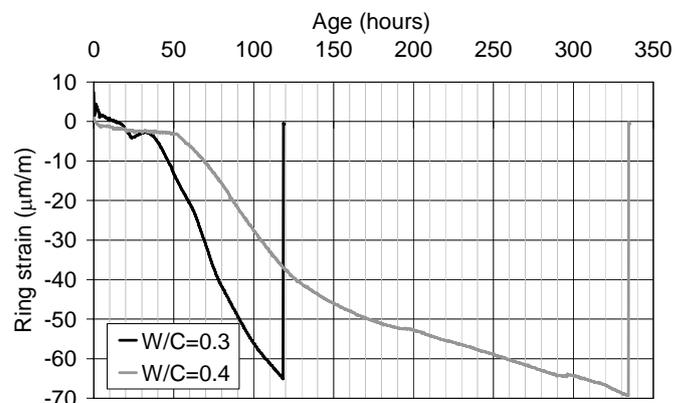


Figure 11. Brass ring strain for cement pastes with $W/C = 0.3$ and $W/C = 0.4$ at $T = 20^\circ\text{C}$.

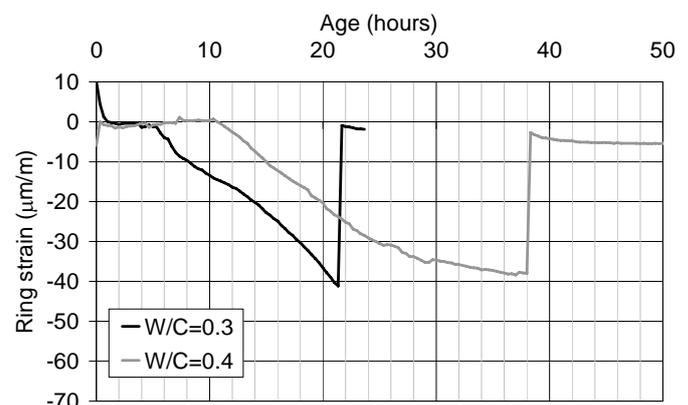


Figure 12. Brass ring strain for cement pastes with $W/C = 0.3$ and $W/C = 0.4$ at $T = 40^\circ\text{C}$.

Autogenous shrinkage is the driving-mechanism leading to early-age cracking, whereas both the rigidity evolution and the tensile strength development of the material are opposed to the increase of these deformations. Previous studies have already shown that a lower water/cement ratio induces an acceleration of the evolution of autogenous deformations (e.g. Baroghel-Bouny & Kheirbek 2001), tensile strength and elastic modulus. The ring strain curves provided in Figures 11 and 12 show that, among the main phenomena involved (autogenous deformation rate, evolution of rigidity and tensile strength development), the preponderant one remains the autogenous strain rate, since the gain of both rigidity and tensile strength due to the diminution of the initial water amount did not enable to slow down the occurrence of early-age cracking.

4 AUTOGENOUS EARLY-AGE CRACKING AND THE HYDRATION DEGREE CONCEPT

The test results discussed in the previous sections are expressed as a function of time. This mode of presentation is not optimal when comparing materials of different compositions since it does not enable to directly relate the cracking age to the physico-chemical evolution of the material.

In the following, the ring test results are plotted as a function of the maturity level of the cement pastes expressed in terms of the hydration degree.

The evolution of the cement hydration degree has been computed with a program developed by the NIST, CEMHYD3D (Bentz 2005).

The input data are the particle size distribution, the chemical composition and the apparent activation energy of cement. For this latter parameter, the value considered is 40 kJ/mol (Mounanga et al. 2006).

Figures 13 and 14 show the influence of the W/C ratio and the curing temperature on the hydration degree of the cement pastes. In the range of the W/C ratio investigated, the effect of this parameter remains weak at a very early age. On the contrary, a significant acceleration of the hydration rate is observed when the temperature increases, in accordance with the thermo-activation principle.

Figures 15 to 18 present the evolution of the ring strain as a function of the cement hydration degree for different cement pastes.

It can be noted that the augmentation of the curing temperature results in a decrease of the maturity level corresponding to the first autogenous cracking time. These results show that, when considering a relatively large temperature range (20 to 40°C), the thermal effect on the first early-age autogenous cracking is not only a kinetic one : temperature influences both the age of cracking and the maturity level corresponding to this cracking age.

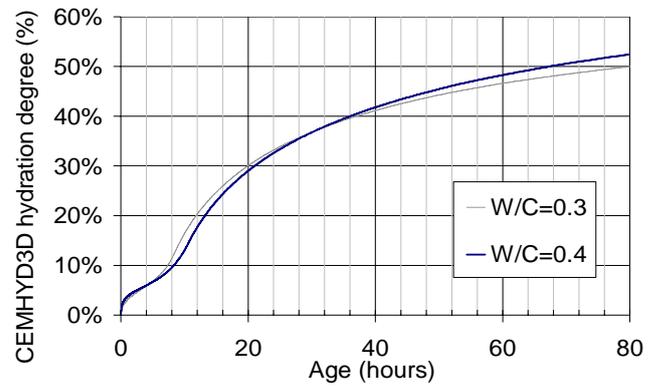


Figure 13. CEMHYD3D hydration degree of cement pastes with W/C = 0.3 and W/C = 0.4 at T = 20°C.

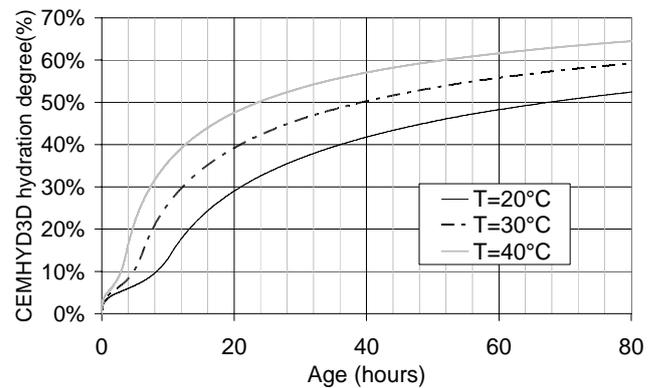


Figure 14. CEMHYD3D hydration degree of cement pastes with W/C = 0.4 at T = 20, 30 and 40°C.

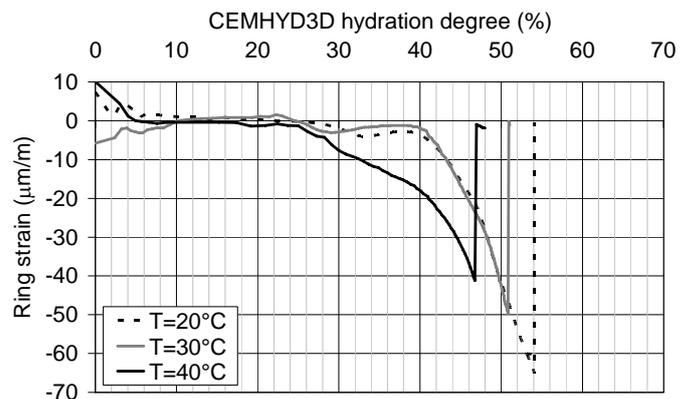


Figure 15. Brass ring test results as a function of hydration degree for cement pastes with W/C = 0.3 at T = 20, 30 and 40°C.

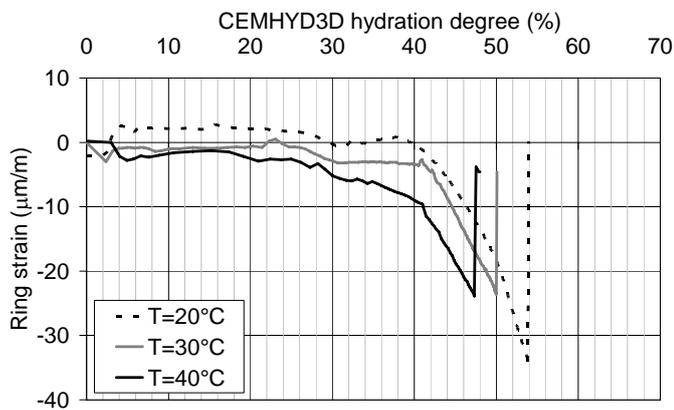


Figure 16. Stainless steel ring test results as a function of hydration degree for cement pastes with $W/C = 0.3$ at $T = 20, 30$ and 40°C .

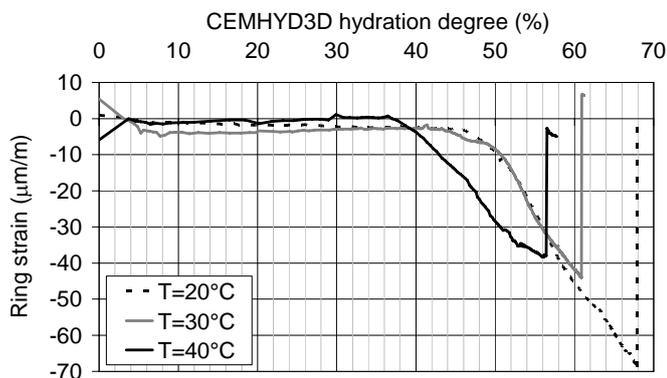


Figure 17. Brass ring test results as a function of hydration degree for cement pastes with $W/C = 0.4$ at $T = 20, 30$ and 40°C .

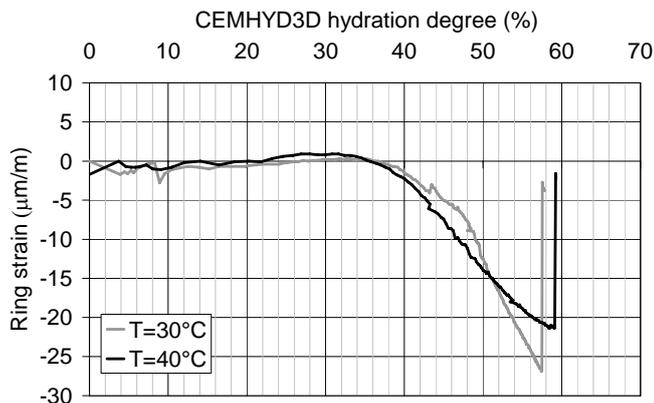


Figure 18. Stainless steel ring test results as a function of hydration degree for cement pastes with $W/C = 0.4$ at $T = 30$ and 40°C .

5 CONCLUSIONS

From the experimental results presented in this paper, the following points can be underlined, concerning:

- *The curing temperature.* A higher curing temperature causes an acceleration of the early age autogenous cracking. This phenomenon

can be attributed to the thermo-activation of hydration, which induces an increase of the autogenous shrinkage rate and therefore of the internal tensile stress. Temperature also influences the maximal amplitude of ring strain recorded just before the cracking.

- *The degree of restraint.* It was observed that the higher the degree of restraint the sooner the autogenous cracking age. Besides, it seems that the surface quality of the ring has an impact on the early age cracking. Supplementary tests are in progress in order to better quantify the effect of this parameter.
- *The water/cement ratio.* A diminution of the initial water amount leads to faster cracking of the cement pastes. This effect has been explained by the fact that, in low water-to-ratio cement-based systems, the autogenous shrinkage rate, responsible for the development of internal tensile stress, is higher.

Finally, from computation results obtained with CEMHYD3D, it was shown that, for a given value of the W/C ratio and in the temperature range studied, the level of maturity (hydration degree) corresponding to the autogenous cracking age decreases when the curing temperature increases.

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