Retention of the mechanical performances of Ductal® specimens kept in various aggressive environments.

Pierre PIMIENTA
CSTB
Champs sur Marne, France

Gilles CHANVILLARD
Lafarge LCR
St Quentin Fallavier, France

Summary

Ultra High Performance Fibre Reinforced Concretes are the kind of materials that can lead to the design of structures in which the fibres take the function of reinforcing bars. This means that the fibres should be able to carry loads even when structural cracks occur. In this context, an essential point for the use of UHPFRC in structural applications is to check their ability to retain the initial mechanical characteristics when the cracked element is subjected to aggressive environment.

In this paper, the results of an experimental study on the evolution of mechanical characteristics of pre-cracked specimens conserved in various conditions are presented. Two types of UHPFRC are evaluated, one reinforced with steel fibres and the other one reinforced with organic fibres. The aggressive environments are hot water, sodium chloride solution and wet-dry cycles. It is shown that the mechanical characteristics of cracked specimens are not affected in such environment.

Keywords: UHPFRC, concrete, steel fibre, organic fibre, mechanical characteristic, flexural strength, aggressive environment, durability

1. Introduction

Ultra High Performance Fibre Reinforced Concretes are the kind of materials that can lead to the design of structures in which the fibres take the function of reinforcing bars. Being inspired by the codes for the prestress concrete and the reinforced concrete, a design code was recently developed in France [1]. This method, based on Serviceability Limit State (SLS) and Ultimate Limit State (ULS), allows to design structures with the classical cracking criteria (non-critical cracking, critical cracking and very critical cracking). In this context, an essential point for the use of UHPFRC in structural applications is to check their ability to retain the initial mechanical characteristics when the cracked structural element is kept in an aggressive environment.

In this paper, the results of an experimental study on the evolution of mechanical characteristics of pre-cracked elements conserved in aggressive environment are presented.

2. General considerations on UHPFRC

Ultra-High Performance Fibre Reinforced Concretes (UHPFRC) have been developed in France since 1991, first with the Reactive Powder Concrete (RPC) [2]. Several other UHPFRC were developed afterwards and today these products are finding many industrial applications. One of them, Ductal® jointly developed by Lafarge, Bouygues and Rhodia, is available as a premix and has been licensed in Japan, France, USA, Canada and Australia[3]. First aimed at ultra-high mechanical performances and ductility [2], these materials also present high durability performances, thanks to their particular microstructure [4,5]. Nine years of accelerated tests have shown that UHPFRC could also be called "Ultra-High-Durability Concretes" (UHDC).

UHPFRC's present high cement content, high amount of ultra-fine components like silica fume and quartz flour which extend the particle size range and small size aggregates or sand. UHPFRC has a very low water cement ratio. It shows a self-levelling behaviour, but a high viscosity. This water to cement ratio is always smaller than the stoichiometric value, required for complete clinker hydration. Therefore in UHPFRC, a large part of the clinker grains (typically in the order of 50%) remains unhydrated [2, 4]. These remaining clinker particles can be considered as surface-reactive micro-aggregates of high elastic modulus, (approx. 120 GPa). They are strongly bonded by low C/S calcium silicate hydrate (CSH) and improve the mechanical performance of the material.

A large part of the high durability of UHPFRC is due to a significant reduction in pore size and volume. The main part of the porosity is found at nanometric scale, within the hydrates nano-structure. Water porosity and permeability arealso close to the detection limit, in relation with the absence of capillary porosity and the disconnected nanopores. These performances are one order of magnitude better than HPC and two orders of magnitude better than ordinary concrete. RPC resistance to corrosion was studied by the CSIC in Madrid (Spain)[6]. After one year of drying and wetting cycles, the metallic fibers in Ductal® FM remain sound, even in a mechanically pre-cracked sample . Only surface stains, approximately at 0.1µm depth, are observed [6].

In the range of Ductal® formulations, two main families can be distinguished. On one hand, thanks to an optimised steel fibre content and a very high performance matrix, a strain hardening behaviour is obtained with this materials under flexural loading leading to a very fine multiple cracking. This range of product could typically be used in structural application where steel fibres can take the function of reinforcing bars according to Interim Recommendation of UHPFRC [1]. On the other hand, the use of organic fibre and again a very high performance matrix allows to obtain a non brittle material for thin sections under flexure. This range of product can be used in architectural applications, where organic fibre can provide the structural integrity in case of cracking [7]. In this paper, the results of an experimental study are presented on the evolution of mechanical characteristics of pre-cracked specimens belonging to previously described families, subjected to various conservation conditions.

3. Experimental program

The formulations of the concretes used in the present study are presented in *Table 1*. For the Ductal® FM, the samples are 70x70x280 mm prisms, notched in the middle for 10 mm, and subjected to 3 point flexural test according to the Interim Recommendations of UHPFRC [1].

Table1 Relative formulations of the concretes

	Sand	**Cement**	**Filler**	**Silica Fume**	**Fibre**	**Fiber (%vol)**	**W/C ratio**
Ductal® FM	1.43	1	0.3	0.325	Steel	2.0	0.20
Ductal® FO	1.43	1	0.24	0.30	APV	4.2	0.27

For the FO formula, the samples are plates of 10 mm x 50 mm x 300 mm subjected to 4 point flexural test according to the NF EN 1170-5 standard, applicable to GRC [8].

Fig. 1 gives an view of the samples preparation, showing the good fluidity and high viscosity of the mixtures, even with high fibre content. 24 Ductal® FM specimens and 9 Ductal® FO plates were cast.

Fig. 1: Overview of the samples preparation

The experimental program includes three main steps. First, after maturity, all specimens were pre-cracked under flexural loading. Then the samples have been unloaded and kept in different aggressive conditions for three months. Finally, the samples have been re-loaded according to the same flexural test procedures as the ones used during pre-cracking, in order to determine the residual mechanical strengths. For each condition, three specimens have been tested.

For Ductal® FM, the maturation followed the following procedure : after casting the material, 48 hours at 20°C and 100%RH, then after demoulding 48 hours at 90°C and 100%RH. For Ductal® FO, a cure of 28 days at 20°C and 100%RH after demoulding was used. This pre-cracking has been done under flexure by adopting displacement control technique. For the Ductal® FM formula, two different crack opening levels have been tested, 100 and 300 µm, both directly measured at the notch location. 100 and 300 µm crack opening levels are considered as the limits in SLS in case of a non critical cracking and very critical cracking states respectively . For the Ductal® FO formula, a cumulative crack opening of 300 µm over a base length of 90 mm in the middle of the plate has been retained. This crack opening has been considered of critical for the structural integrity of a thin element, which looks extremely damaged at that state. Then, the various aggressive environments used are:

- **Reference samples:** they are kept at 23°C and 50% relative humidity.
- **Hot water:** the samples have been kept immersed into water at 60°C
- **Wet-dry cycles:** The tests have been conducted according to the EN 494 Standard, either in water at 20°C for 18 hours and then 6 hours of drying at 60°C (60 cycles in total).
- **Chemical environment:** Continuous immersion into a 10% sodium chloride solution. (this test has only been done for Ductal® FM).

In addition, during these tests, resonance frequency and mass of the samples have been regularly measured.

4. Results and discussion

4.1. Retention of mechanical properties

For each configuration, three samples were tested. The deviation between them is small and then, only one curve will be presented, representing the mean behaviour. Each curve displays flexural stress, calculated with the applied load by considering the linear classical mechanics formulas in function of crack opening displacement.

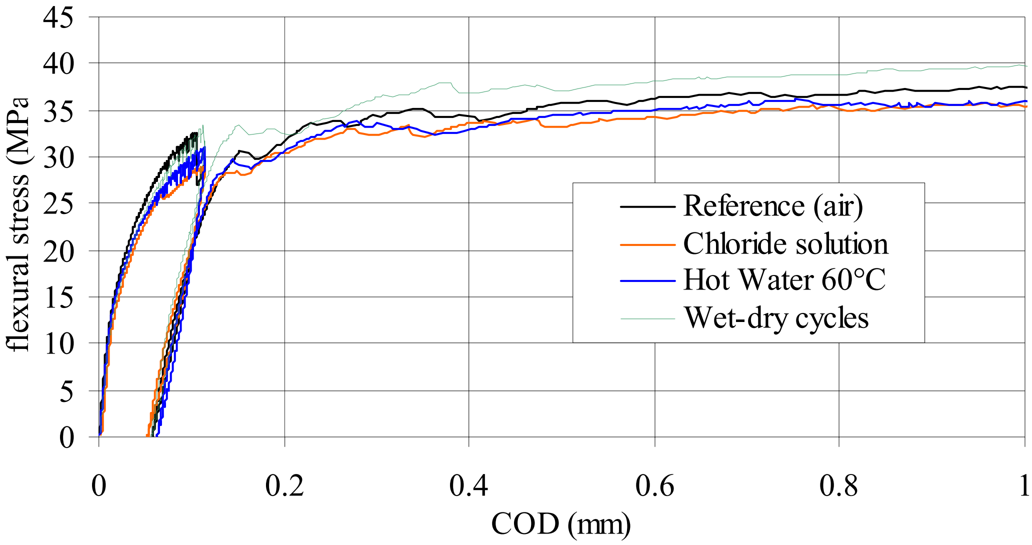


Fig.2 : Flexural Stress vs. COD curve for Ductal® FM, pre-cracking 100 µm

Figures 2 to 4 give the synthesis of the obtained results. The pre-cracking stage appears clearly as the first part of each curve and the re-loading corresponds to the second part. This two stage of loading have been connecting for the purpose of the presentation. In practice, the data acquisition was not set at the same rate in both loading stages, so that the noise on the curve could be different. However, the control rate of the test was kept constant at 60µm/min in both stages.

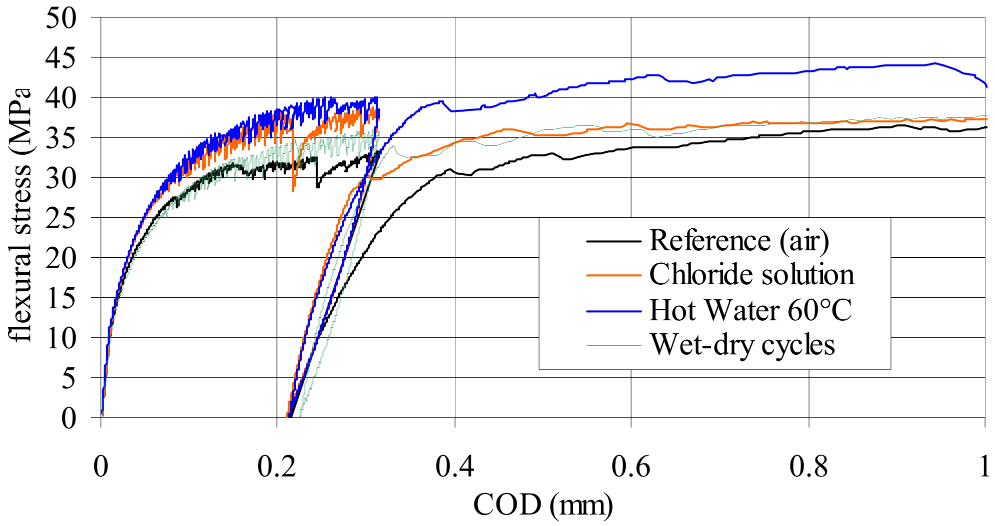


Fig.3: Flexural Stress vs. COD curve for Ductal® FM, pre-cracking 300 µm

Then, it appears clearly that the mechanical characteristics of the cracked samples have not been influenced by the severe conditions of conservation used. In case of Ductal® FM (fig. 2 and 3) the continuity of all the curves is remarkable. The only point we can identify is the shape of the reloading curve for 300 µm pre-cracking in reference configuration. The slope of this curve is less stiff than in the other conservation cases. We can also notice that the reference configuration is the only one without contact of the cracked surface with water. Mechanisms of self sealing may be responsible of such behaviour, it will be discussed later in this paper.

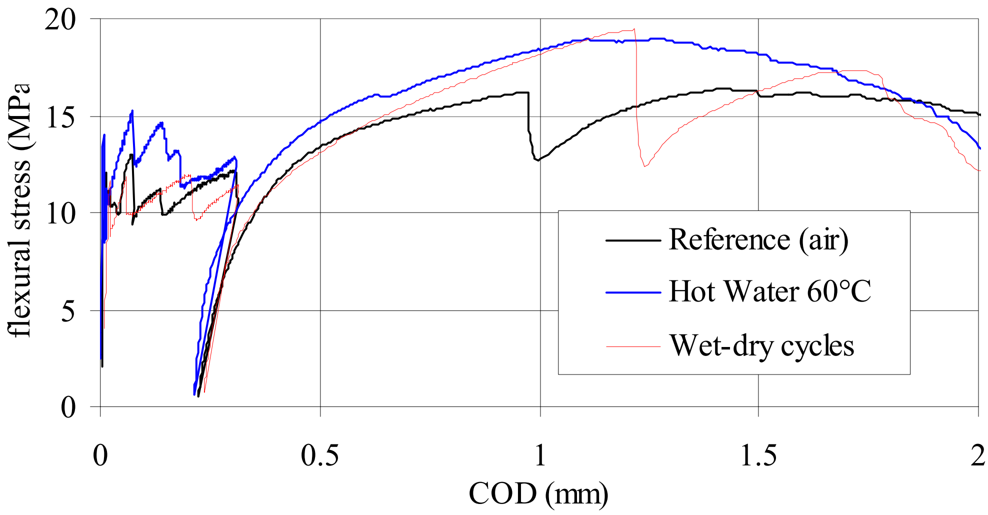


Fig.4: Flexural Stress vs. COD curve for Ductal® FO, pre-cracking 300 µm

In the case of Ductal® FO, the same conclusion can be drawn. It is interesting to note that precise load drops can easily be identified on these curves (fig. 4). Each of these drops correspond to the localization of a new crack in the central part of the specimen under flexure. In addition, even after conservation in aggressive environment, the fibres bridge the cracks at a sufficient level so that a new crack is formed.

4.2. Resonance frequency and mass evolutions

Considering now the evolutions of resonance frequency and mass. For the reference environment, no notable evolution is measured on both measures. For the other environments, the evolution of the mass is still very small and it does not exceeded 0.5%. The reliability of the resonance frequency allows a more interesting analysis (fig. 5).

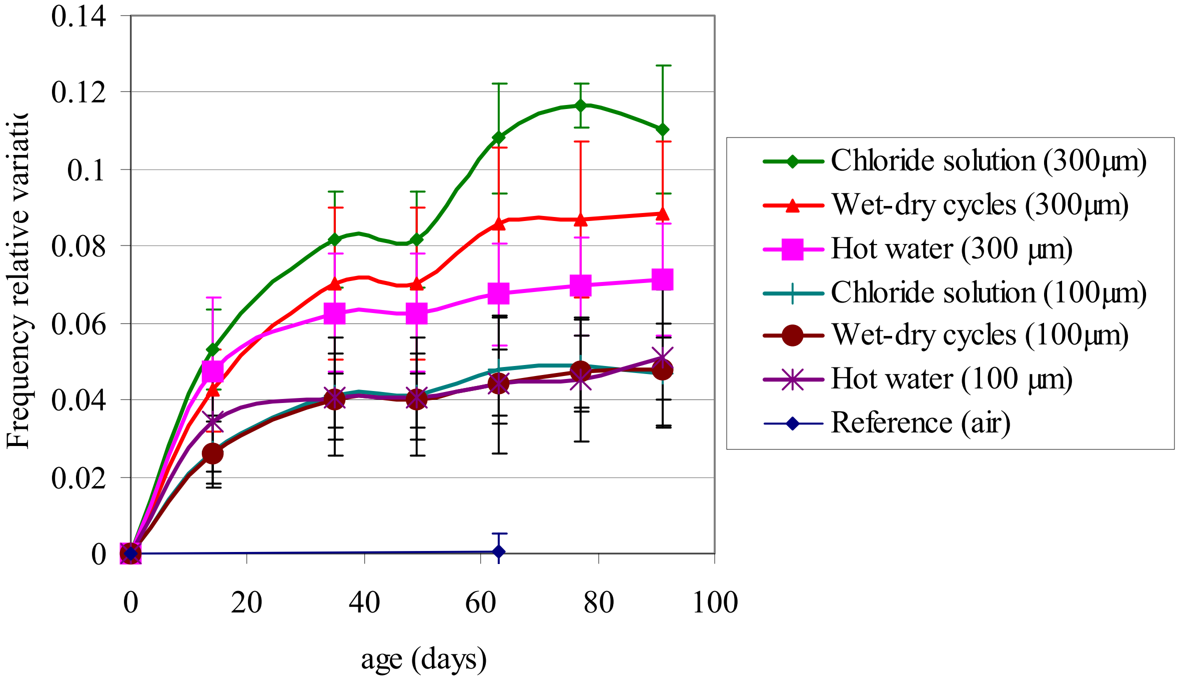
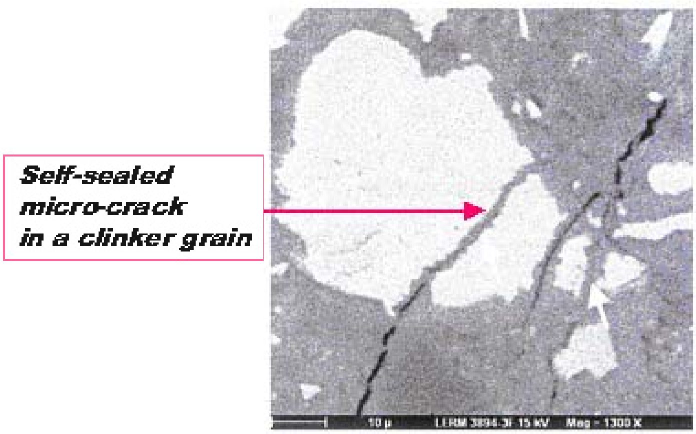


Fig.5: Resonance Frequency vs. age for Ductal® FM

For Ductal® FM, it appears that the resonance frequency increases with duration of tests, showing a gradual stabilisation. In addition, this increase in higher for larger initial crack opening. It seams that any distinction between the various aggressive environment is not possible considering the standard deviation of the measure. However, it is well known that the resonance frequency is directly correlated with the dynamic elastic modulus of the material. Then, starting with a cracked sample, any increase in the resonance frequency indicates that the overall rigidity of the samples has increased.



One possible explanation of such phenomena is the self sealing of the microcracks. Indeed, we have already mentioned that a large part of the cement remains unhydrated in UHPC. Then, microcracks may open paths for the water which allows additional hydration of the cement. Such behaviour has already been observed in a previous study dealing with durability of the microstructure (fig. 6). Opening cracks of less than 40 µm were completely filled by hydration products after conservation under water [9].

Fig. 6 : Self-sealing of microcracks by clinker hydration

As a consequence, the resonance frequency may be more sensitive in the case of 300µm crack opening than 100µm because the numbers of paths created during pre-cracking is larger. This is confirmed by the initial mean values of the resonance frequency (before conservation protocols), which are 3.46kHz (Sdt Dev 0.05kHZ) for Ductal® FM 100µm and 3.18kHZ (Sdt Dev 0.1kHZ) for Ductal® FM 300µm. Finally, such self sealing doesn't exist in case of conservation in air (the reference state). This may explain the shape of the reference curve compared to the others on fig. 3 and the previous consideration explaining why such effect is not visible on fig. 2.

Finally, this hypothesis on self sealing should be confirmed in the present study by additional works on the tested specimens, using SEM.

5. Conclusion

In this paper, the results of an experimental study on the evolution of mechanical characteristics of pre-cracked Ductal® specimens based on steel fibre reinforced and organic fibre reinforced formulas are presented. Various conservation conditions have been tested, including hot water, wet-dry cycles and sodium chloride solution.

After three months, it appears that the mechanical characteristics of the cracked samples have not been influenced by the severe conditions of conservation used. Then this retention of the mechanical properties shows that such material may be used in structural applications as far as the crack opening is less than 300µm. Possible self sealing of the microcracks has been proposed considering the evolution of resonance frequency. This point should be confirmed with further observations using SEM of the specimens' microstructure.

6. References

[1] AFGC, "Ultra High Performance Fibre-Reinforced Concretes, Interim Recommendations", AFGC Publication, France, January 2002

[2] RICHARD, P. and CHEREZY, M. "Les bétons de poudres réactives*" Annales de l'ITBTP*, N°532, Mars-Avril 1995, 1995, pp. 83-102.

[3] CASANOVA, P., DUGAT, J., and ORANGE, G., "Ductal : a new generation of Ultra-High Performance Fiber Reinforced Concrete" *Proc. HPC Symp., Workability, Strength and Durability*, Hong Kong, December 10-15, 2000. Vol. 2, pp. 853-859, C.K.Y. Leung, Zongjin Li, Jian-Dong Ding Editors.

[4] VERNET, C., LUKASIK, J., and PRAT, E., "Nanostructure, permeability, and diffusivity of Ultra-High Performance Concretes (UHPC*)". International Symposium on High Performance and Reactive Powder Concretes,* Sherbrooke, Québec,1998, Vol.3, pp.7-36.

[5] VERNET, C., "UHPC Microstructure and related Durability Performances : Laboratory Assessment and Field Experience Examples" *PCI / FHWA 3rd International Symposium on HPC, Orlando*, Florida, 19-22 Oct. 2003

[6] ROUX, N., ANDRADE, C., and SANJUAN, M.A, "Experimental Study of Durability of Reactive Powder Concretes" *Journal of Materials in Civil Engineering,* Feb. 1996, pp. 1-6.

[7] PERRY, V. and ZAKARIASEN, D., "An Overview of UHPC (Ductal®) Technology, Materials, Properties, Markets and Manufacturing" *PCI / FHWA 3rd International Symposium on HPC Orlando*, Florida, 19-22 Oct. 2003

[8] NF EN 1170-5 "Precast concrete products – test method for glass-fibre reinforced cement – part 5 Measuring bending strength" Standard AFNOR, Mai 1998

[9] AMMOUCHE, A. and MARTINET, G., "Durability of Ductal® and ancient BPR" Rapport d'étude n° 98.3894.003.01.A, LERM, August 2001