

**Durability of fibre reinforced cementitious composites
Coupling mechanical and chloride environment loads**

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DURABILITY OF FIBRE REINFORCED CEMENTITIOUS COMPOSITES: COUPLING MECHANICAL AND CHLORIDE ENVIRONMENT LOADS

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Abstract

Fibre reinforced cementitious composites (FRCC) may be characterized by their improved performance namely in terms of tensile ductility, accompanied by multiple cracking, and potentially lower permeability to liquid and gas in cracked state. Cracking, which is nearly inevitable, can occur due to applied structural loading, shrinkage, chemical attack, thermal deformations and restrained condition. Even though might not be a structural problem, cracking could be a durability issue, since it considerably modifies the transport properties of the cementitious composite and, as consequence, accelerates the deterioration process, which can significantly impair the long term service life of a structure or element. Literature indicates that, particularly for chloride penetration, the presence of cracks and/or load condition, causes a more deleterious attack compared to standard durability test on sound specimens composites. Contributing to that concern a methodology for accessing chloride attack in loaded and/or cracked FRCC is proposed. Cracking procedure and specimen geometry were selected, considering that cracks produced in laboratory should resemble those in structural elements (beams). Thus, FRCC specimens were firstly pre-loaded under four point bending up to a pre-defined crack width. The crack width was kept using special stainless steel frame. In addition, cracked but not loaded specimens were considered and sound specimens were used as reference. Then, specimens were exposed to wet-dry cycles in a chloride solution. It is argued that the chloride penetration is definitively influenced by the load and cracking conditions, which promoted a higher penetration depth leading to a severe fibres corrosion, which also compromised the mechanical performance of FRCC.

1. INTROUDCTION

More than 50% of Europe's annual construction budget is spent on rehabilitation and refurbishing of deteriorated concrete structures [1]. Indeed, the cost of maintenance of bridges in Europe exceeds 1 billion euros per year [2]. Thus, it is argued that new fibre reinforced cementitious materials might be used to rehabilitate reinforced concrete exposed to aggressive environments, aiming to improve their overall durability performance [3].

Over the last decades, considerable efforts to improve the behaviour of cementitious materials have led to the emergence of Ultra-High Performance Fibre Reinforced Cementitious Composites (UHPRFC). These novel building materials provide the structural engineer with an unique combination of extremely low permeability, which prevents the ingress of detrimental substances, and very high strength, namely, compressive strength

higher than 150 MPa, tensile strength over 7 MPa and with considerable tensile strain hardening (3–10%) and softening behaviour. These outstanding properties are promoted by an ultra-compact cementitious matrix incorporating a considerable volume (usually > 2%) of high strength short steel fibres. Considering the using of UHPFRC in rehabilitation/strengthening applications, in which the UHPFRC layer will be subjected to aggressive environment and/or high mechanical load (e.g. concentrated forces, wear, fatigue, impact), the final objective of the present research was to perform a more realistic durability evaluation. Thus, simulating real field scenarios (XS3/XD3) and quantify how cracking and/or loading condition influences the permeability and damage of UHPFRC to aggressive agents, namely, chloride penetration is the final objective to pursue.

The current paper reports on a preliminary research carried out at TU-Delft (Microlab), during an internship of the first author. The main objective of the internship was to implement a methodology and procedure which allows determination of the effect of combined mechanical and environmental actions on fibre reinforced cementitious composites. In that context, a methodology for accessing chloride attack in loaded and/or cracked cementitious composites materials is proposed. A special frame was designed to simulate a load condition keeping a predefined open crack width in the specimens. Then, specimens were kept simultaneously in the frame and in an aggressive environment (wet-dry cycles employing chloride aqueous solution), thus coupling a mechanical and environmental load. Given the time limitations of the referred internship, it was decided to perform the preliminary tests using a very porous matrix (water/binder=0.26; water/cement=1.73) produced with local available materials.

2. FRAME DESIGN

Most structural elements in real structures are subjected to bending, which origins V-shape (flexure) cracks as assumed in the codes. A four-point bending test, considering the proportional dimensions and loading scheme of Figure 1, was employed. A stainless-steel frame to maintain a predefined crack width in a four-point bending load system was built to carry out the durability tests (see Figure 3-b). The specimen's dimensions were (40x40x160) mm³. Due to the small dimensions these specimens are easy to cast, transport and require only a small volume of material.

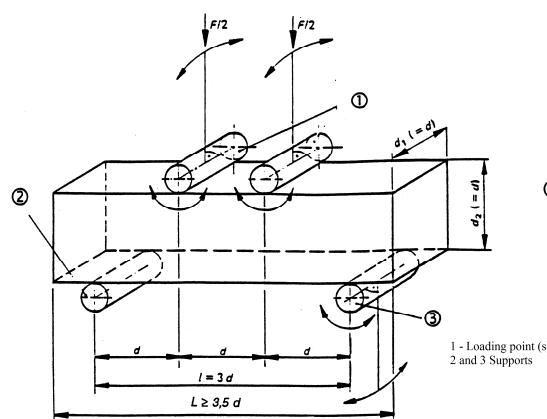


Figure 1: Schematic view of four point bending loading scheme [4].

3. EXPERIMENTAL PROGRAMME

3.1 Materials, specimen's production and test methods

The materials used in this study were: Portland cement Type I 42.5 N complying requirements of EN 197-1 [5], ground granulated blast furnace slag (GGBS) in accordance with EN 15167-1 [6], limestone filler, siliceous sand (1 mm maximum aggregate size, density of 2660 kg/m³ and 0.04% water absorption), superplasticizer (density of 1080 kg/m³ and 32% solid content) and micro steel fibres with 10 mm length and 0.20 mm diameter (tensile strength of 2750 MPa and Young Modulus of 200 GPa).

The mixtures were prepared in batches of 3L using a Hobart two-speed mixer in accordance with the EN 196-1 [7]. Table 1 and Table 2 present the mixture proportions and the mixing procedure, respectively. The FRCC fresh state properties were characterized using the mini-slump flow test performed as recommended by Okamura et al. [8] (Dflow). After the fresh state test, prismatic specimens (40x40x160) mm³ were prepared following the procedure prescribed by the standard EN 196-1 [7] except the compaction step. Specimens were demoulded in the following day and then were cured in controlled environment chamber at (20±2)°C and RH≥95% until they reach the testing age for assessment of the mechanical properties and for cracking before the initiation of the durability test. The flexure and compressive strength tests were performed at 9, 36 and 180 days (36 days correspond to the age of cracking and 180 days corresponds to end of chloride exposure) following the procedure described in the standard EN 196-1 [7] as can be seen in Figure 2. In addition, setting time was measured in the mixture without short steel fibres and following the procedure of EN 480-2 [9]. The mixture properties in fresh, hardening and hardened states are presented in Table 2.

3.2 Specimens cracking methodology

Codes establishes limitation of crack width in order to, namely, keep the good visual appearance of the structure and water proofing performance and to prevent rebar reinforcement corrosion. The maximum crack widths allowed by different codes for aggressive environmental conditions vary approximately between 0.15 and 0.30mm. A crack width between 0.20-0.30mm was set as the target in the present study. Predefined crack opening displacement (COD) of approximately 300µm, was produced through four-point bending loading. The test was carried out by means of an Instron 8872, equipped with a 10kN load cell, controlling the deflection speed (displacement rate = 0.01mm/s). (Figure 3-a). Two linear variable differential transducers (LVDT) were fixed on each side of the specimen, perpendicular to loading direction, to monitor the COD. When predefined COD was achieved the specimens were unloaded and taken out from the Instron machine. Keeping the LVDTs on the specimens they were immediately allocated on the stainless-steel frame. Subsequently, the specimens were re-loaded up to the predefined COD using threaded rods tightened by a torque wrench. Preserving the LVDTs on the specimen allowed achieving the desired COD again. Afterwards, LVDTs were carefully removed and the frame sustained the COD. as is shown in Figure 3-b. These specimens were referred as Frame,300µm. In addition, cracked specimens with residual crack (not kept in the frame) were considered (Res,_{250µm}). Sound

specimens (not cracked-Ref,_{0μm}) were also used as reference and control specimens were maintained in the curing room during all test period (CTL). Five specimens were considered for each condition. A typical load-average COD curve is depicted in Figure 4, in which the first part of the curve includes elastic deformation, followed by the occurrence of non-linearity, and finally unloading when the specimen crack partially closes up to 50μm. Afterwards, all specimens were dried during 7 days in a controlled environment room (20°C and RH=(50±5)%).

Table 1: Mixing sequence.

Task	Duration	Speed
Add cement + slag + limestone filler + fine aggregate + fibres	2.5 min	140 ± 5 rotations·min ⁻¹
Add water + superplasticizer while mixing	1.0min	140 ± 5 rotations·min ⁻¹
Mix	1.0min	140 ± 5 rotations·min ⁻¹
Mix	4.0min	285 ± 10 rotations·min ⁻¹

Table 2: FRCC mixture proportions and mixture properties in fresh, hardening and hardened states.

FRCC mix composition constituents (kg/m ³)	Binder (b)	Cement (c)	183,72
		Blast furnace slag (bfs)	425,00
		Limestone Filler (f) (kg/m ³)	612,40
	Aggregates	Sand	608,72
	Water (w)		317,82
	Superplasticizer (Sp)		2,20
	Fibres		117,50
FRCC mix composition (main) ratios	w/b		0,26
	V _f (%)		1,50
FRCC characterization	Fresh state	Dflow (mm)	250
	Setting time (EN 480-2)	Initial (hh:mm)	04:12
		Final (hh:mm)	06:17
	Compressive strength (EN 196-1)	R _{c,9d} (MPa)	47±1.8
		R _{c,36d} (MPa)	58±1.9
		R _{c,180d} (MPa)	68±1.8
	Flexure strength (EN 196-1)	R _{f,9d} (MPa)	19±1.9
R _{f,36d} (MPa)		20±2.0	
R _{f,180d} (MPa)		32±0	



Figure 2: Determination of strength at 9, 36 and 180 days according to EN 196-1 [7]: a) flexure strength (3 point bending); b) compressive strength.

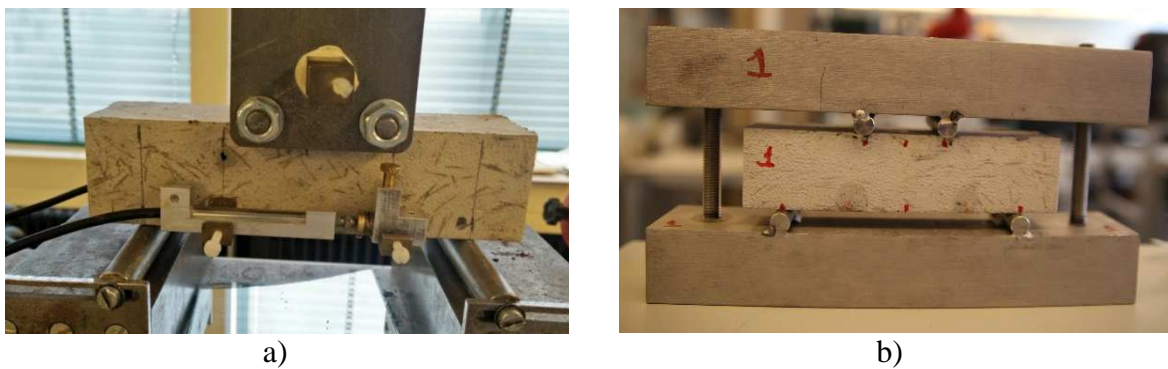


Figure 3: Frame: a) Beam specimen in Instron machine while loading was applied; b) Crack width kept by the frame.

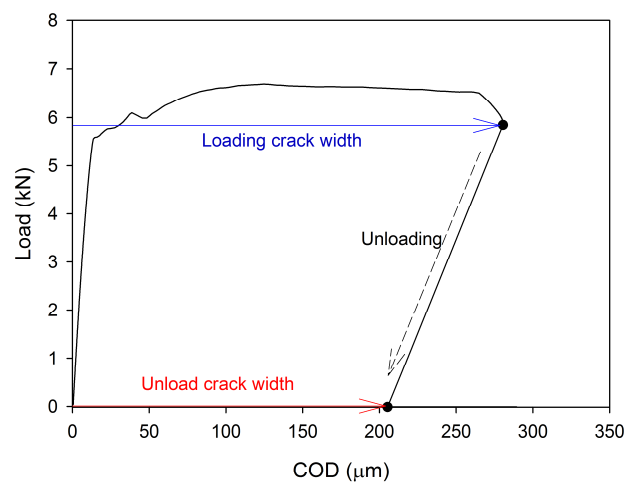


Figure 4: Typical load-displacement curve for 4 bending point loading.

Long term experiment for chloride penetration

For the long-term experiment with chlorides exposure, framed, cracked and virgin specimens were used. The side surfaces of all prisms were sealed to ensure the chloride penetration tough cracked surface. Then, specimens were partial immersed in an artificial chloride solution, simulating sea water (3.5% NaCl), and undertaking weekly wet-dry cycles consisting of 48 hours partial immersion and 5 days of drying at 20°C and 50%RH. The chloride bath was covered in order to avoid as much as possible the solution evaporation. The level of the solution in the container was measured once a week and filled if needed.

4. RESULTS AND DISCUSSION

4.1 Chloride penetration analysis

At the end of 20 wet-dry cycles, it was generally observed the fibre corrosion on the exposed surface, an example of that is shown in Figure 5-a). Using a Microscope Multizoom Nikon AZ100 it was possible to observe in detail the fibre corrosion, as well as, the corrosion products, see Figure 5-b). In addition, micro-cracking due to the fibre corrosion was found, as depicted in Figure 6. For fibre cementitious composites, it is inevitable that some of the fibres will be very close to the surface. Thus, these have an almost negligible, cover depth. This should make fibres especially susceptible to external agents and, consequently, high degradation would be expected for these fibres when exposed to extreme environments. Previous research experimentally confirmed that fibres located at depths up to 3 mm suffered severe corrosion whereas the rest of the fibres, i.e. those fully embedded inside of the concrete, remained free from corrosion [10], [11].

Specimens were dry cut longitudinally in two parts with a diamond saw. After removing powder on the specimen surface, a 0.1M silver nitrate solution was sprayed on one of the cut sections. The chloride penetration depth can then be measured from the visible white silver chloride precipitation. A border between the chloride contaminated area and non-contaminated was found, when compared with typical boundary obtained in accelerated tests, see Figure 7. This can be explained by the slowly entrance of chloride, not forced by an electric potential difference, as well as, by the lower concentration of chloride solution used (3.5% NaCl in the present long term test and 10%NaCl in accelerated test according NT Build 492 [12]). For cracked specimens, two measures of chloride penetration depth can be drawn, the penetration in unfractured zone (D_m) and in the fractured zone (D_{crack}), see Figure 7-b), as presented in Table 3: Chloride penetration depth in unfractured (D_m) and fractured zone (D_{crack}).

Specimens	D_m (mm)	D_{crack} (mm)
Ref, ₀ μm	6.8	-
Res, ₂₅₀ μm	6.9	33
Frame, ₃₀₀ μm	6.0	33

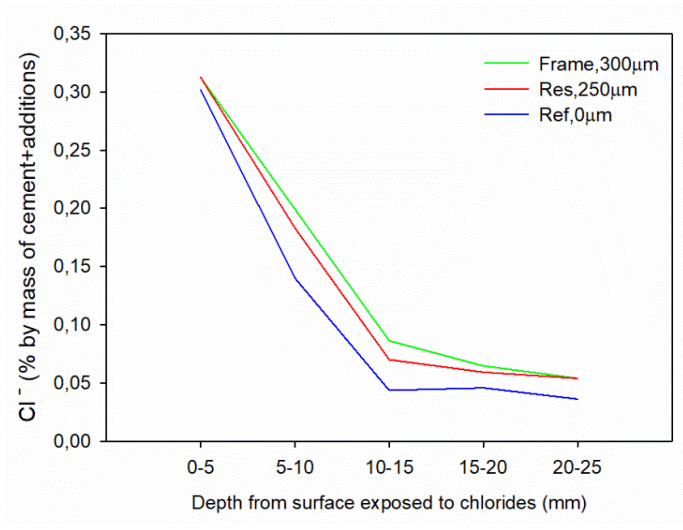
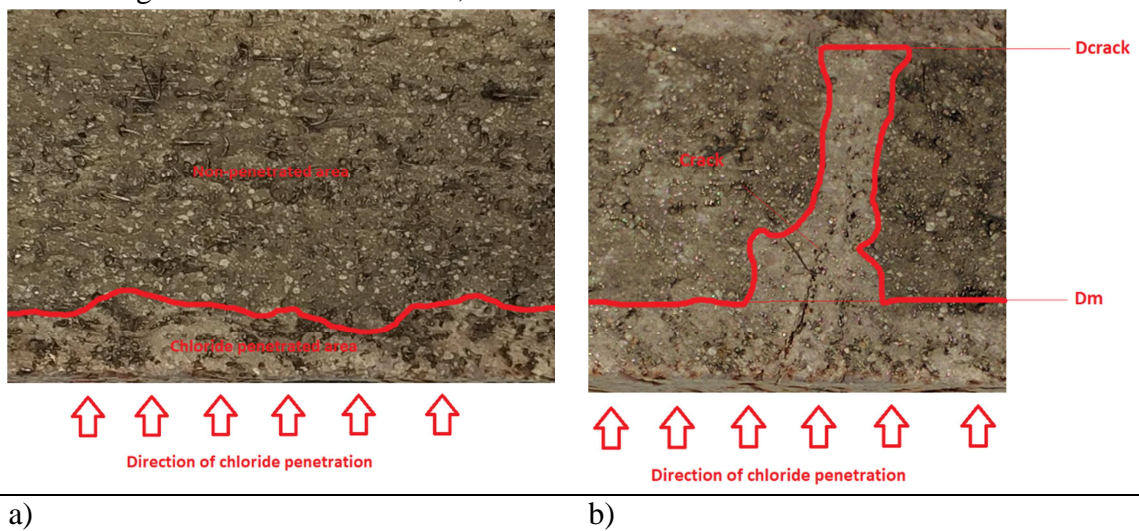


Figure 8: Chlorides profile.

In the cracked specimens, a localized and significant chloride penetration (30mm) was observed in the fractured zone, while in reference specimens an uniform penetration has occurred.

The other half of cut specimens was used for chemical analysis (chloride content). The samples were taken from the central part of the specimen (in between the two load points) and in the direction of chloride penetration. The “dry drilling method” sampling procedure, according to RILEM Recommendation TC 178-TMC [13] was employed to obtain powdered samples (removing short steel fibres) corresponding to depth steps of approximately 5 mm. The chloride profiles are presented in Figure 7. The chloride content is expressed as the percentage of chloride ions by mass of cement, or the total amount of binder if any supplementary materials is added [14]. This value shall not exceed the boundaries established on the Table 15 from EN 206-1 [14], where the critical chloride content generally accepted is within the range of 0.2-0.4% of Cl^- , for conventional reinforced concrete structures.



a) Reference specimen and b) cracked specimen.

According with the EN 260-1 Portuguese Annex [15], for exposure class XS/XD, chloride threshold value is 0.2%. The reference specimens showed a high chloride concentration, close to the specimen's surface, in agreement with the colorimetric analysis with silver nitrate, and then a decrease of chloride content is observed. This can be explained by the slowly progress of chloride penetration which is governed by diffusion in the simulation of natural conditions test. On the other hand, in cracked specimens, the chloride penetrated inwards rapidly due to the induced crack promoting an easy entrance for the ions. Critical chloride contents are observed up to 10 mm from the specimens' surface. Although in colorimetric analysis the chloride penetration depth was similar in cracked specimens, Res_{,250µm} and Frame_{,300µm}, the chloride content is higher in Frame_{,300µm}. It seems, since crack was forced to keep open, the chloride could ingress in higher amount. Based in the previous findings, it is argued that the chloride penetration is influenced by the load and cracking conditions, which promoted a higher penetration depth and chlorides concentration.



Figure 5: a) Exposed specimen surface after wet-dry cycles; b) Detail of fibre corrosion on specimen surface.

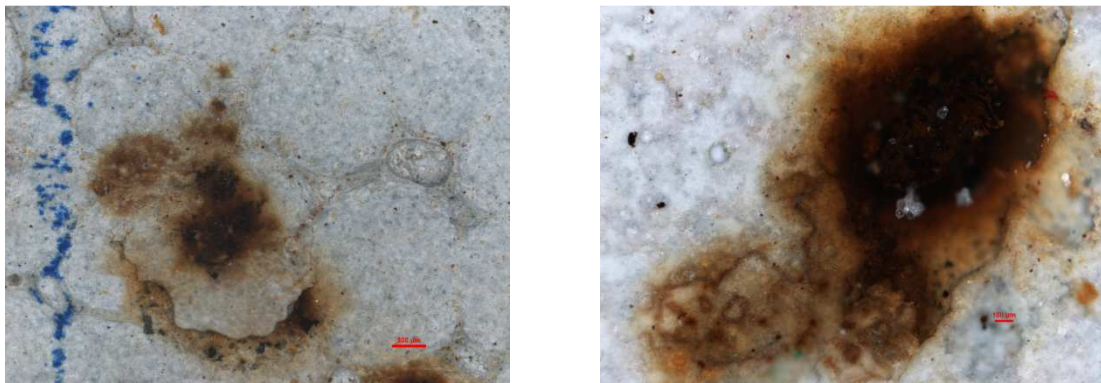


Figure 6: Micro-cracking due to fibre corrosion.

Table 3: Chloride penetration depth in unfractured (D_m) and fractured zone (D_{crack}).

Specimens	D_m (mm)	D_{crack} (mm)
Ref, _{0µm}	6.8	-
Res, _{250µm}	6.9	33
Frame, _{300µm}	6.0	33

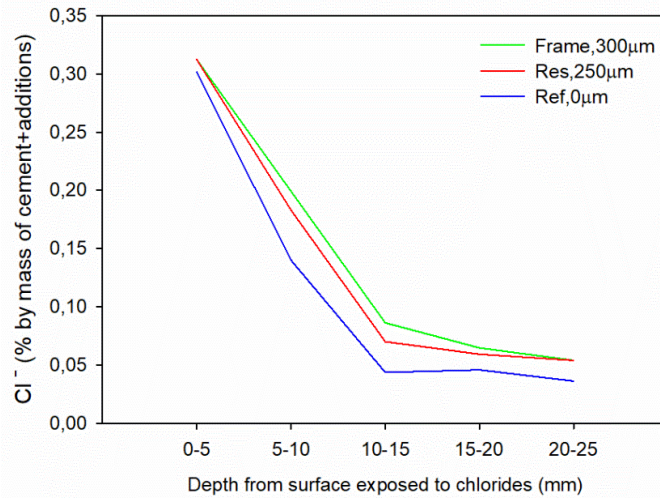


Figure 8: Chlorides profile.

4.2 Mechanical performance after chloride exposure

After wet-dry cycles, the flexure and compressive strength were accessed (according EN 196-1-Figure 2) on chloride exposed specimens, as well as, on specimens kept in curing room all the stage (CTL). Results are presented in Figure 10 and it can be observed a significant decrease in flexure behaviour, namely on cracked and loaded specimens (Frame,300µm), in which approximately 50% of flexure strength was compromised. A significant recovery of these specimens would not be expected due to the continuous damage (crack) during chloride exposure. In cracked specimens (Res,250µm) the flexure behaviour was not affected in the same level, and performed equivalent to reference specimens (not cracked-Ref,0µm). Some crack closure due to the CaCO₃ precipitation (autogenous healing) or latter hydration from the matrix might have occurred. Concerning compressive strength slighter deleterious effect was noticed presenting losses above 10 MPa maximum, comparing CTL and Frame,300µm

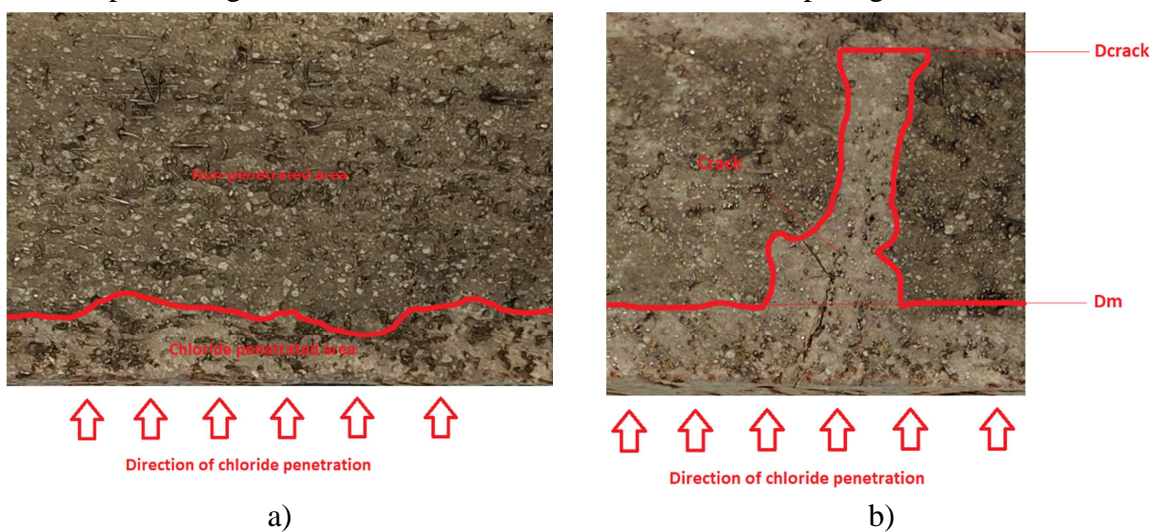


Figure 7: Chloride penetration profiles on: a) Reference specimen and b) cracked specimen.

specimens. Blast furnace slag presents hydraulic activity (slow “hydration” ratio), which provide a continuously development of the degree of hydration when incorporated in cement based materials. Consequently, an enhancement on compressive strength is achieved by Ref,_{0μm}. After splitting the specimens it was possible to visualize the chloride effect on previous cracked cross section for Frame,_{300μm} and Res,_{250μm}, as well as, on Ref,_{0μm} specimens, as shown in Figure 9. A serious damage due to chloride penetration was observed, namely for Frame,_{300μm} specimens, in which fibre corrosion achieved almost 20mm. This phenomenon explains the loss of flexure strength. Concerning Res,_{250μm}, considerable chloride penetration was also notice, however lower than the Frame,_{300μm}, giving rise to a lower deleterious action on flexure behaviour. Ref,_{0μm} present lower chloride penetration, though also compromising the bending performance.



Figure 9: Corrosion of short steel fibres after wet-dry cycles.

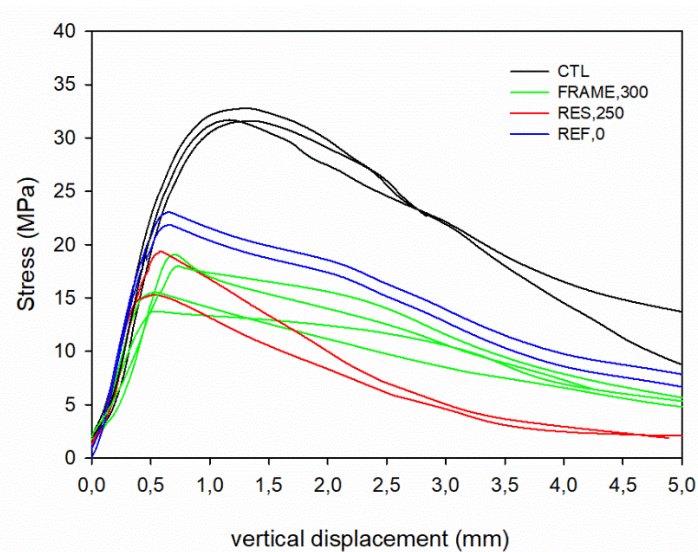


Figure 10: Flexure strength of CTL, reference (Ref,_{0μm}), cracked (Res,_{250μm}), and cracked and loaded specimens (Frame,_{300μm}).

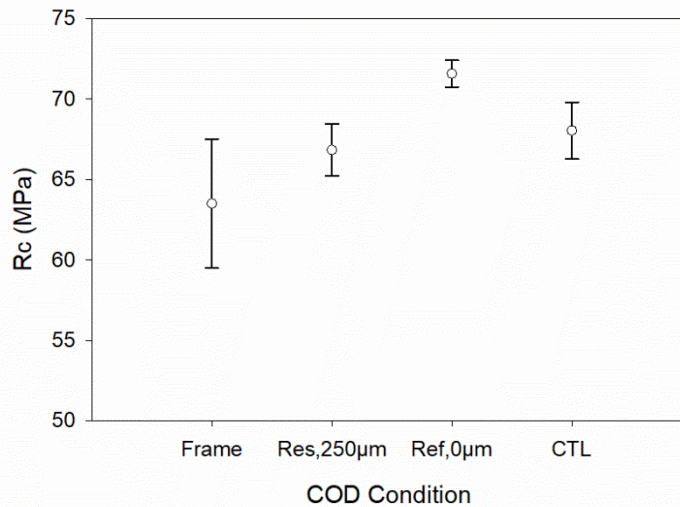


Figure 11: Compressive strength of CTL, reference (Ref, $0\mu\text{m}$), cracked (Res, $250\mu\text{m}$), and cracked and loaded specimens (Frame, $300\mu\text{m}$).

5. CONCLUSIONS

The existence of cracks is one of the main causes of rapid deterioration of structural concrete members under chloride-laden environment. In this study an experimental program was conducted to examine the effect of a crack width (suggested as admissible on codes) and loading condition on the chloride penetration. The results presented in this paper contribute a step forward on the durability design coupling mechanical and environmental loads, in which could be concluded:

- Visual examination of non-cracked specimens after long-term exposure showed absence of corrosion in fibres, which were embedded within the concrete. On the other hand, in cracked specimens fibre corrosion was observed in fracture zones.
- The cracks act as a bridge connecting the external environment to the internal parts of the concrete. A penetration depth of approximately 30mm was found on the crack's surrounding area. This indicates that the load and cracking conditions were determinant to allow easy chloride ingress.
- The chemical analysis revealed chlorides content beyond critical boundaries suggested by the codes. The chloride content was higher only on measurements close to the surface (0-5mm).
- Damage of mechanical properties (flexure) occurred on cracked FRCC specimens after 20 cycles of exposure, which can be explained by the serious chloride ingress providing the fibre corrosion. In reference specimens, flexure performance was also compromised in a lighter level.

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