

## **EFFECT OF NATURAL FIBRES ON THE SELF-HEALING CAPACITY OF HIGH PERFORMANCE FIBRE REINFORCED CEMENTITIOUS COMPOSITES**

**Liberato Ferrara (1), Saulo Rocha Ferreira (2), Visar Krelani (1), Flavio Silva (2,3) and Romildo Dias Toledo Filho (2)**

(1) Department of Civil and Environmental Engineering, Politecnico di Milano, Italy

(2) Civil Engineering Department, COPPE, Universidade Federal do Rio de Janeiro, Brasil

(3) Department of Civil Engineering, Pontificia Universidade Catolica - Rio de Janeiro, Brasil

### **Abstract**

In this paper the effect of natural fibres on the self healing capacity of cementitious composites has been studied. Natural fibres, through their porous micro-structure, can create a large number of moisture paths, through which moisture, absorbed by the same fibres, can be released “on demand” and activate delayed hydration processes which contribute to the healing of the cracks. Reference has been herein made to a High Performance Cementitious Composite reinforced either with steel fibres only or with a hybrid mix of steel and sisal fibres. Influence of environmental conditions has also been studied, including exposure in a climate chamber with high humidity (90% RH), low humidity (50% RH), wet and dry cycles and immersion in water has been performed. The effects of self healing on the recovery, if any, of mechanical performance has been quantified by means of mechanical performance in bending (residual stresses at prescribed openings, stiffness etc.). Visual stereoscopic observation will be instrumental at visualizing the healed cracks and also assess the amount of crack closure to be correlated to the recovery of mechanical properties, evaluated as above.

### **1. INTRODUCTION**

The demand for high level service and performance structures and infrastructures is increasing, and is dictating the need to associate to high mechanical performance also enhanced durability and minimum negative ecological impact.

Self-healing materials can provide interesting and effective solutions to these problems.

As a matter of fact, even in most traditional concrete mixtures, a certain percentage of the cement remains not hydrated even after long time. The greater the fineness of the cement and the lower the water/cement ratio of the mixture, the higher is the percentage of non hydrated cement. When cracking occurs, the cement grains that have not reacted may come in contact

with the moisture that penetrates into the cracks and a delayed hydration process can be activated and the products of hydration can not only fill the crack, “sealing” it, but may also contribute to some recovery of mechanical properties and hence to an overall “healing” of the material and structure performance. In the case of High Performance Fibre Reinforced Cementitious Composites (HPFRCCs), the behaviour of which is characterized by a stable process of formation of multiple tiny cracks, the matrix composition, featuring high content of cement and binders showing either cementitious and/or pozzolanic activity and a low water binder ratio, and the tight opening of each single crack, are likely to provide a great potentiality for autogenous healing.

In this framework, the use of natural fibres in HPFRCCs is likely to be characterized by a twofold value. On the one hand they can contribute to the multiple cracking behaviour, which, together with a peculiar mix composition, is an essential requisite for conductivity to autogenous healing, as remarked above. On the other hand because of their highly hydrophilic nature and porous microstructures, natural fibres, either even simply during the mixing process or through dedicated pre-saturation, can absorb water. This water, through the porous network consisting of the same dispersed natural fibre reinforcement, can be released “on demand” at selected cracked or damaged locations and activate the delayed hydration reactions which are responsible of the healing processes of damage and cracks.

In the framework of project EnCoRe this problem has been investigated with reference to a HPFRCC, reinforced with steel fibres and with a hybrid steel-sisal combination (in equal volume percentages). In this paper, an experimental methodology will be proposed to evaluate the effects of crack healing on the mechanical properties of HPFRCC, with reference to their fibre-orientation dependent behaviour. The proposed methodology (Ferrara and Krelani, 2013; Ferrara et al., 2014) consists in pre-cracking, up to different crack openings beam specimens by means of a displacement-controlled four point bending (4pb) set-up. The influence of the age of concrete at pre-cracking was also considered. Specimens were then subjected to different exposure conditions, as detailed in the forthcoming section, and, at the end of scheduled exposure, times 4pb tests were performed (again) on the same specimens. Results, in terms of load-COD curves, were compared with those obtained from virgin specimens, in order to evaluate recovery of mechanical properties, if any, in terms of load bearing capacity, stiffness, damage accumulation and ductility. The cross comparison between steel and hybrid fibre reinforced composite will allow the effectiveness of natural fibres in promoting crack healing process to be assessed.

## **2. EXPERIMENTAL PROGRAM: MATERIALS AND TESTS**

The cementitious matrix, in which the different types of steel and natural fiber reinforcement were incorporated, consisted of a mortar made of cement, ground granulated blast furnace slag, sand sieved to a maximum diameter of 2 mm (the granulometric curve is shown in Figure 1), water and a polycarboxylate super-plasticiser (Glenium 51; solid content 31.20%). Portland cement type 52.5 R (rapid hardening), with the composition listed in Table 1 was employed. The chemical composition, as from XRF spectrometry, of the employed slag (particle size class 12  $\mu\text{m}$ ) is summarized in Table 2. The mixtures were produced in a room with controlled temperature ( $21 \pm 1^\circ\text{C}$ ) using a mixer with capacity of 20 l and following the mixing protocol in Table 3. A mixture reinforced with steel and sisal fibres (SS) and a control mix with only steel fibres ST, were prepared. The mix proportions are given in Table 4.

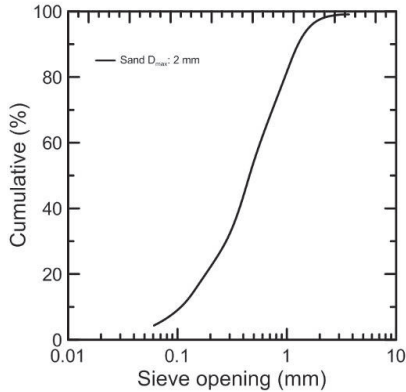


Figure 1: Sand grain size distribution

Table 1: Chemical composition of cement.

C <sub>3</sub> S	65%
C <sub>2</sub> S	11%
C <sub>3</sub> A	9%
C <sub>4</sub> AF	6%

Table 2: Chemical composition of GGBS

SiO <sub>2</sub>	39.0 %
Al <sub>2</sub> O <sub>3</sub>	11.0 %
Fe <sub>2</sub> O <sub>3</sub>	0.70 %
TiO <sub>2</sub>	0.55 %
CaO	37.50 %
MgO	8.30 %
K <sub>2</sub> O	0.30 %
Na <sub>2</sub> O	0.20 %
MnO	0.80 %
C	0.20 %
S	1.00 %

Table 3: adopted mixing protocol

Mix raw cement and slag at 50 rpm	1 m
Add water and SP	1 min
Mix paste at 50 rpm	5 min
Add sand while mixing at 50 rpm	2 min
Mix mortar at 100 rpm	5 min
Add fibers while mixing at 100 rpm	2 min 30 sec
Mix FRCC at 100 rpm	5 min

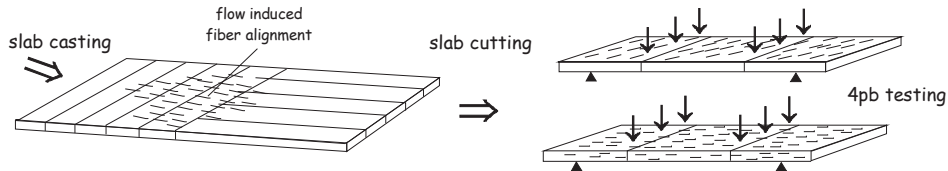
Table 4: mix proportions of investigated HPRCC mixes

Constituent	Dosage (kg/m <sup>3</sup> )	
	HPFRCC ST*	HPFRCC SS
Cement type I 52.5	600	600
Slag	500	500
Water	200	200
Superplasticizer	33 (l/m <sup>3</sup> )	40
Sand 0-2 mm	1000	1000
Sisal fibres	-	70
Steel fibres	100	50

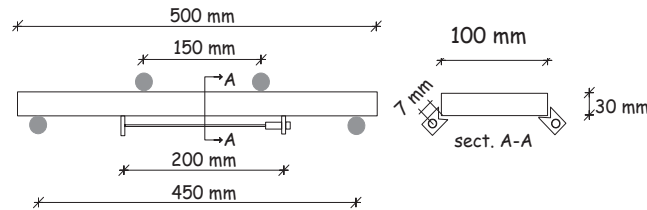
Table 5: Characterization of sisal fibers

Fiber type	Density (kg/dm <sup>3</sup> )	Cellulose (%)	Hemicellulose (%)	Lignine (%)	Main length (mm)	Sect.area (mm <sup>2</sup> )
Sisal	1.01	60.5	25.7	12.1	13	0.023

Sisal fibres were obtained from sisal plants cultivated in farms located in the Bahia state, Brazil. They were extracted from the sisal plant leaves in the form of long fibre bundles. The fibre extraction from the leaf was done by semi-automatic crushers. From 10kg of sisal leaves about 1.35 kg extractable fibres could be obtained. These fibres were characterized mechanically by Silva et al (2009; 2010). The main characteristics of the fibres employed in this study are summarized in Table 5. High carbon straight steel fibres, 13 mm long and with a diameter equal to 0.16 mm, produced by Bekaert (OL 13/0.16) were employed. As from the manufacturers, the minimum tensile strength of the wire is equal to 2000 MPa. Fibres come with a surface brass coating.



**Figure 2:** slab casting scheme and beam specimen cutting procedure

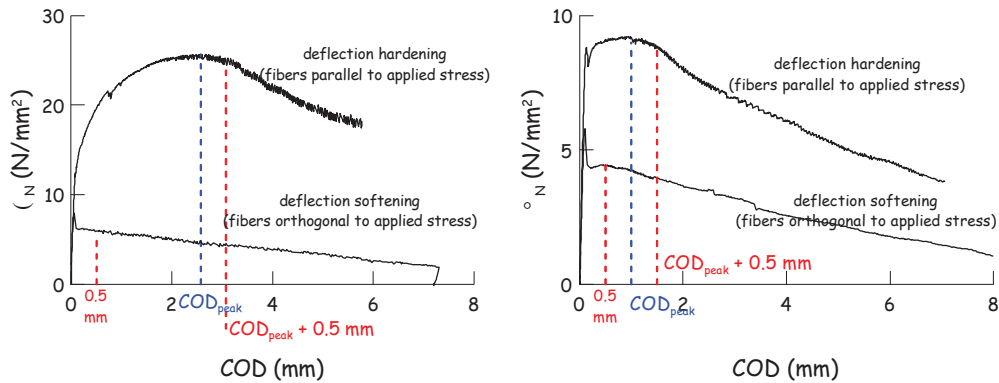


**Figure 3:** 4-point bending tests set-up for beam specimens obtained as in Figure 1.

With each mix, slabs 1m long x 0.5 m wide x 0.03 m thick were cast. The employed casting process consisted in pouring the fluid mixture at one short edge of the mould; the mix, because of its self levelling abilities, was able to flow up to the opposite edge, completely filling the formwork and triggering the alignment of the fibres along the casting flow direction. After 24 hours, the specimens were de-moulded and placed in a fog room and moist cured for 28 days. From each plate, after 15 days curing in fog room, ten beam specimens were cut, 500 mm long and about 90 mm wide, using a circular saw (Figure 2).

After about 45 more days in the fog room, all specimens were pre-cracked adopting a 4-point bending test set-up, as shown in Figures 3; tests were performed with an electromechanical testing machine. The adopted testing configuration is in accordance with Italian guidelines CNR-DT 204, which recommend the characterization of the material through test on un-notched specimens in the case of thin elements (with a height less than 150 mm), and/or with a hardening type behaviour, which both apply to the present study. In this way it is also possible to take into account the direction of the casting and possible flow induced alignment of the fibres. Tests were displacement controlled, at a rate equal to 5  $\mu\text{m}/\text{sec}$  and the crack opening (COD - Crack opening Displacement), was also measured by means of two LVDT transducers (Figure 3) over a gauge length equal to 200 mm.

In order to calibrate the target pre-cracking value of the Crack Opening Displacement, monotonic tests up to failure were performed, employed the set-up described above, for each of the fibre reinforced mixes under investigation, taking into duly account the alignment between the flow induced orientation of the fibres and the direction of the applied stress (Figures 4-5). Specimens featuring a deflection softening response (i.e. with fibers perpendicular to the beam axis) were pre-cracked up to a COD value equal to 0.5 mm whereas for specimens with fibers parallel to the axis, most likely featuring a deflection hardening response, in order to work under the assumption of the same opening of the localized crack, the specimens were pre-cracked in the post-peak regime equal to  $(\text{COD}_{\text{peak}} + 0.5 \text{ mm})$ , where  $\text{COD}_{\text{peak}}$  denotes the measured value of the COD in correspondence of the peak stress.



**Figure 4:** monotonic response of steel HPFRCC specimens for fibres parallel and orthogonal to the applied bending stress – COD pre-cracking thresholds are highlighted. **Figure 5:** monotonic response of steel+sisal HPFRCC specimens for fibres parallel and orthogonal to the applied bending stress – COD pre-cracking thresholds are highlighted.

After pre-cracking specimens were “conditioned” in four different “environments”:

- immersion in water (wet);
- dry (in a chamber with 20°C temperature and 50% relative humidity);
- moist (in a chamber with 20°C temperature and 90% relative humidity);
- wet and dry cycles: the cycles were made by alternating one day of immersion of specimens in water, at a temperature of 20 ° C, and one day of exposure in a dry environment at a temperature of 20 ° C and 50% relative humidity.

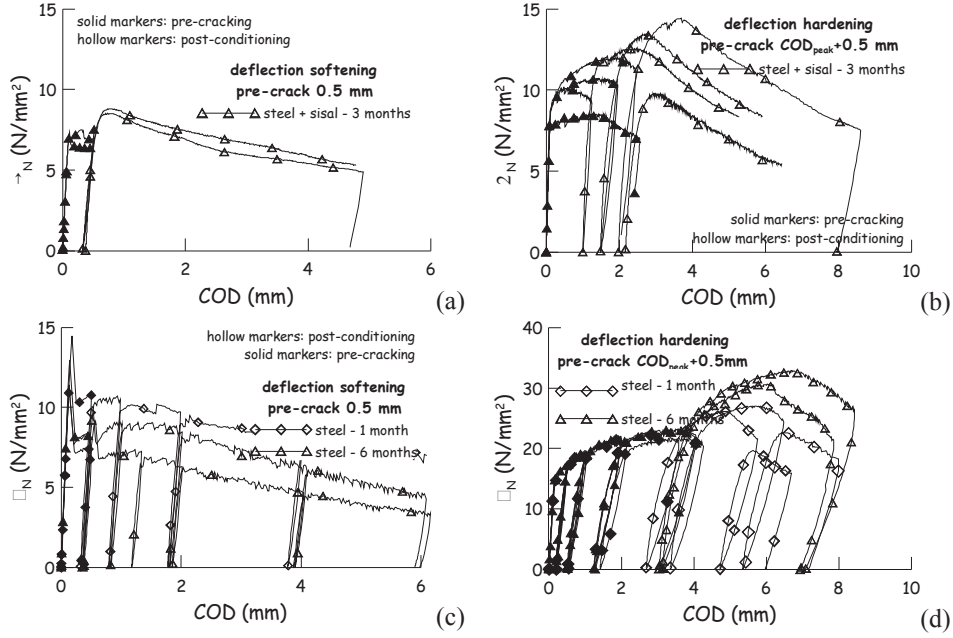
At the end of the scheduled conditioning period, specimens were tested up to failure according to the same 4-point bending set-up shown in Figure 3. “Superposition” between pre-cracking and post-conditioning  $\sigma_N$ -COD curves allowed self healing capacity and its effects on mechanical performance of the material to be evaluated. Results shown in this paper refers to the early stage of the investigation, which is still on-going.

### 3. EXPERIMENTAL RESULTS

In Figure 6 the nominal stress  $\sigma_N$  vs. COD curves are shown for deflection softening and hardening specimens, reinforced with both steel and hybrid steel+sisal fibres, with reference to wet-and-dry cycles exposure conditions, for which the effect of natural fibers, able to absorb water and release it to surrounding matrix during drying was more evident.

For deflection softening specimens the effect of crack healing is evident from the attained new peak stress, higher than the residual stress at unloading, as well as for a more gentle softening measured in post-conditioning stage, which, in the case of prolonged exposure times, can even resemble a quite stable deflection hardening behaviour.

The effects of self healing are in case remarkable also for deflection hardening specimens pre-cracked beyond the peak COD value, as witnessed by their capacity, at least for the shown exposure conditions, to attain, in the post-conditioning stage, a new peak, even higher than the one that featured the virgin specimen behaviour in the pre-cracking stage, as well as by the capacity to retain a deflection hardening behaviour.



**Figure 6:**  $\sigma_N$  vs. COD curves for deflection softening (a,c) and deflection hardening (b,d) steel+sisal (a,b) and steel (c,d) specimens, for wet and dry cycles.

Comparison in terms of nominal bending stress  $\sigma_N$  vs. COD curves, referring to the same specimen in the pre-cracking and post-exposure stages, will allow the effects of self healing to be evaluated; comparative evaluation between steel and hybrid fibre reinforced composites will also be performed, including effect of exposure time on the kinetics of the crack healing.

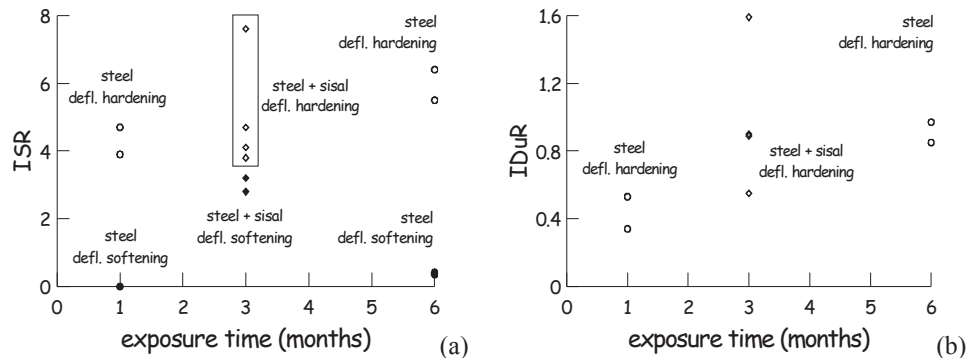
### 3.1 Self healing indices

From the curves plotted in Figures 6, a to h and qualitatively discussed above, a quantitative evaluation of the effects of self healing phenomena will be as hereafter performed, following a procedure already developed for self healing of ordinary concrete (Ferrara et al., 2014 a-b). For deflection softening specimens the “stress gain” due to self healing is evaluated as in Eq. (1) whereas for deflection hardening specimens a self healing stress gain index and an index of ductility recovery were calculated, as in (Eq.2) and (Eq. 3):

$$\text{Index of stress recovery ISR} = \frac{f_{\text{peak,post-treatment}} - \sigma_{\text{unloading}}}{f_{\text{ft,pre-cracking}} - \sigma_{\text{unloading}}} \quad (1)$$

$$\text{Index of stress recovery ISR} = \frac{f_{\text{peak,post-treatment}} - \sigma_{\text{unloading}}}{f_{\text{peak,pre-cracking}} - \sigma_{\text{unloading}}} \quad (2)$$

$$\text{Index of ductility recovery IDuR} = \frac{\text{COD}_{\text{peak,post-treatment}} - \text{COD}_{\text{unloading}}}{\text{COD}_{\text{peak,virgin specimen}}} \quad (3)$$



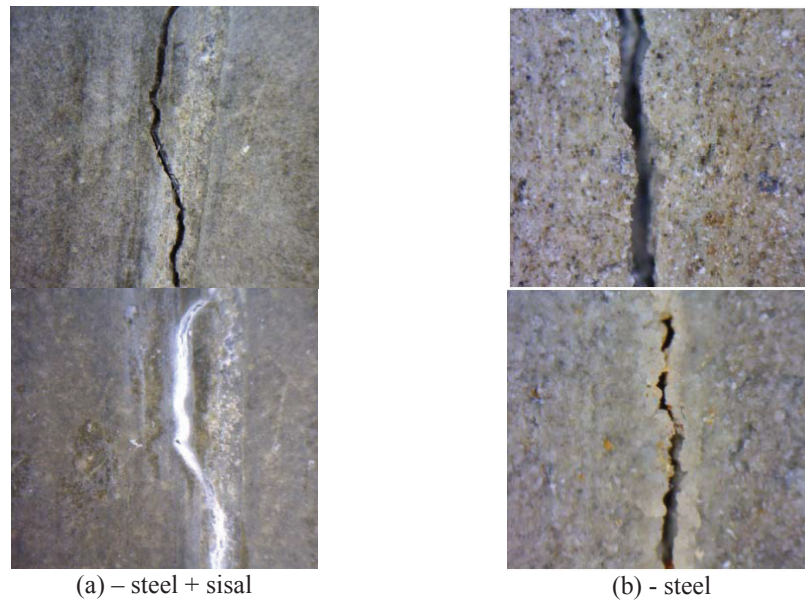
**Figure 7:** index of stress (a) and of ductility recovery (b) for different analyzed specimens.

The Indices related to self healing capacity are plotted in Figures 7 a-b.

It can be interestingly observed that, whereas deflection softening specimens reinforced with steel fibres only did exhibit negligible stress recovery capacity, either upon short or long term exposure, deflection softening specimens reinforced with a hybrid mix of steel and sisal fibres, even after three months cycling, exhibited a significant capacity of recovery the stress bearing capacity lost upon pre-cracking softening stage (see also Figure 6a). On the other hand, deflection hardening specimens, even when pre-cracked in the post-peak stage, exhibited a significant capacity of recovering both the stress and the ductility; for steel fibre reinforced specimens, for which evolution data along the exposure cycles were available, both the aforementioned properties positively evolved along the cycling time. this can be reasonably explained considering the narrowest opening of each single crack which forms in the stable multi-cracking process which characterizes the behaviour of deflection hardening specimens. It also significantly appear that, even after only three months cycling, specimens reinforced with steel and sisal fibres were able to provide a recovery, either in terms of stress or ductility, quantitatively comparable to the one exhibited by steel fibres upon a cycling time twice as much longer. In the authors' opinion this could be rightly attributed to the natural fibres, which, even if not pre-saturated before mixing, were able to absorb water during the wet phase of each cycle and gradually release it, also to the surrounding concrete matrix where un-reacted cement and slag particles are available and able to capture it. In this way the delayed hydration reactions which are responsible of the crack healing can be more effectively promoted. Example of visually evident healed cracks are shown in Figure 8, for specimens reinforced with either type of fibre reinforcement.

#### 4. CONCLUSIONS

In this paper the preliminary results have been shown of an experimental campaign devoted at assessing the effectiveness of natural fibres, when hybridized with other kind of fibre reinforcement, such as steel, to promote self healing reactions in cement based materials, due to their hydrophylic nature. The results shown, despite partial and referring also to the initial stage of the investigation, are likely to confirm the aforementioned assumption. Research is ongoing also to evaluate the effects of different scale of natural fibre reinforcement, including micro-fibres from different sources and cellulose nano-pulps.



**Figure 8:** completely healed cracks in steel+sisal (a) FRC deflection softening specimens after 3 months cycling and of partially healed cracks in steel (b) FRC deflection softening specimens after 6 months cycling (0.5 mm pre-crack; different scales of magnification).

#### ACKNOWLEDGEMENTS

This work was mainly developed during in the months between July 2013 and May 2014, as a part of the activities carried out by the authors within the “EnCoRe” Project (FP7-PEOPLE-2011-IRSES n. 295283; [www.encore-fp7.unisa.it](http://www.encore-fp7.unisa.it)) funded by the European Union within the 7<sup>th</sup> Framework Programme. Particularly it began during the second and third author secondment between Politecnico di Milano (Italy) and Universidade Federal do Rio de Janeiro (Brasil). The contribution of Mr. Marco Della Torre and Mr. Gregorio Sanchez Arevalo in performing experimental tests, in partial fulfilment of the requirements for the MEng degree in Building and Civil Engineering respectively is also gratefully acknowledged.

#### REFERENCES

- [1] Silva, F.A., Mobasher, B. and Toledo Filho R.D., “Cracking mechanisms in durable sisal reinforced cement composites”, *Cement and Concrete Composites*, **31** (2009) 721-730.
- [2] Silva, F.A., Toledo Filho R.D., Melo Filho, F.A. and Fairbairn, E.M.R., “Physical and mechanical properties of durable sisal fiber cement composites”, *Construction and Building Materials*, **24** (2010) 777-785.
- [3] Ferrara, L., Krelani, V. and Carsana, M., “A “fracture testing” based approach to assess crack healing of concrete with and without crystalline admixtures”, *Construction and Building Materials*, **68** (2014) 535-551 .
- [4] Ferrara, L., Krelani, V., Geminiani, M. and Gorlezza, R.: “Self- healing of high performance fiber reinforced cementitious composites”, in Proceedings fib2014 World fib Symposium, Mumbai, India, 10-14 February 2014, 883-887.