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A FRACTURE TESTING BASED APPROACH TO ASSESS THE SELF HEALING CAPACITY OF CEMENTITIOUS COMPOSITES

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Abstract: The self healing capacity of cementitious composites employed for either building new or repairing existing structures opens challenging perspectives for the use of a material intrinsically able to recover its pristine durability levels, thus guaranteeing a longer service life of the designed applications and a performance less sensitive to environmental induced degradation. One possibility of achieving the aforementioned self-healing capacity stands in the use of additives featuring a "delayed crystalline" activity, which, when in contact with water or atmosphere humidity, form chemical compounds which are able to reseal the cracks thus guaranteeing the recovery of a pristine level of mechanical performance. In order to quantify this self healing ability, either in presence of not of the aforementioned additives, and its effects on the recovery of mechanical properties of concrete a methodology has been developed and will be presented in this paper: it allows the recovery of material properties to be evaluated in terms of stiffness, maximum load and effective crack opening and "self healing" indices to be defined and quantified in a "durability based" design framework.

1 INTRODUCTION

Civil and structural engineers have to face with continuously increasing urgency multifaceted problems dictated by the increasing demand of structures and infrastructures in developing countries and by the aging of existing ones in the "developed" world. The rapidly changing society needs may not seldom require the aforementioned facilities to be built and used in extreme climate and/or service conditions, which poses a high demand to the structural performance. Because of concrete cracking and deterioration over time, in order to satisfy the aforementioned requirements all along the structure service life, the initial performance of concrete has to be set at a quite high level, which results in increased construction costs. Furthermore, the need to apply comprehensive maintenance systems has to be likewise foreseen, which, though able to extend the service life of the structure and retrieving, partially or completely, the pristine levels of performance, will also present the drawbacks of additional costs.

Worldwide increasing consciousness for sustainable use of natural resources has made "overcoming the apparent contradictory requirements of low cost and high performance a challenging task" [1] as well as a major concern. The availability of self-healing technologies, by controlling and repairing "early-stage cracks in concrete structures were possible" [1], could on one hand prevent "permeation of driving factors for deterioration" [1], thus extending the structure service life, and, on the other, even provide, in case, partial recovery of engineering properties relevant to the application.

As pointed out by Lauer and Slate [2], as early as in 1956, in their milestone study on autogenous healing of cement paste, “if the mechanism of the *[self healing]* action is understood, and means can be found for accelerating it, a great stride will have been made in effectively retarding the rate of the disintegration of concrete, *which […]* is one of the major problems of the concrete field […]”.

Path has been travelled since then and consensus among the international community has been achieved about the engineering significance of the problem, which has resulted in state-of-the-art reports to be compiled as well as into a clear terminology definition.

The RILEM TC-221-SHC “Self healing phenomena in cement based materials”, reported by [1], distinguishes:

- based on the result of the action, between self-closing and self-healing, whether only closure of the cracks or also restoring of the properties is observed;
- based on the process of the action, between “autogenic” (or natural) and “autonomic” (or engineered), whether the crack closure or restoration of material properties is due to either own concrete material or some engineered addition.

In this paper, a new approach based on fracture testing to quantify the effects of natural or engineered self healing will be presented and discussed, also focusing the attention on the use of crystalline admixtures as catalysts of the self healing phenomena.

2 “A CONCRETE MIRACLE?”

Autogenous healing of cement based materials was reportedly discovered as early as in 1836 by the French Academy of Sciences, and attributed to the convection of calcium hydroxide exuded from the hydrated cement and converted into calcium carbonate on exposure to the atmosphere. Such a discovery was reported by Loving [3] who, on inspection of concrete pipe culverts in 1936, found many healed cracks filled with calcium carbonate.

Already in 1913, Abrams [4] observed the phenomenon and attributed it to the retarded or interrupted hydraulicity of the cement. Further investigations followed [1], through which it was recognized the nature of the phenomenon to be that of continued hydration. As a matter of fact, Turner [5] pointed out that the action of autogeneous healing has “practical value in several applications […] namely , the increase in strength of partially set concrete remixed with water, repair of precast units cracked during early handling; sealing against corrosion and re-knitting of cracks developed in concrete piles during their handling and driving; sealing of cracks in concrete water tanks; and the regain, after loss, of strength of “green” concrete disturbed by vibrations”.

Further evidence of the phenomenon was reported by Whitehurst [6], who observed an increase in the dynamic modulus of field structures during a wet spring, following a winter of freezing and thawing, correlating it to at least some improvement in the strength properties of the concrete.

Lauer and Slate [2] finally provided the first comprehensive investigation and explanation of the self healing mechanisms. As a matter of fact that the materials produced by the self-healing reactions consist of calcium hydroxide and calcium carbonate crystals. The latter are due to the reaction between calcium hydroxide, which is a product of cement hydration, and carbon dioxide present either in water or air. The consumption of calcium hydroxide on the crack surfaces generates its outward migration from inner concrete. At the same time, as long as the production of calcium carbonate continues, its crystals precipitate along the free surfaces of the crack. They found direct correlation between the size of the crystals, the percentage of surface crack area covered by crystals and the healing strength. Furthermore they found healing in an atmosphere at 95% relative humidity could give up healing strengths as much as 85% lower than those obtained by healing under water, for which up to 25% of the normal 90 days strength could be recovered upon cracking at 1 day. Obviously, the later the cracking occurred, the lower the percentage of healed to normal strength.

The importance of continuing hydration as a driver for the self-healing capacity of cementitious composites was further highlighted by Hearn and Morley [7]. Continuing or delayed hydration, as explained by Neville [8], most likely occurs in early age concretes, where cracks are more tortuous, because of the lower strength and toughness of paste, and may thus expose to outdoor environment larger clusters of anhydrous cement particles. On the other hand, in old concretes the material filling the cracks mainly consists of calcium carbonate, according to the aforementioned reaction mechanisms.

Summarizing previous studies possible mechanisms of self-healing can be categorized as follows [9]:

(1) further reaction of unhydrated cement;
(2) expansion of the concrete in the crack flanks;
(3) crystallization of calcium carbonate;
(4) closing of the cracks by solid matters in the water;
(5) closing of the cracks by spalling-off of loose concrete particles resulting from the cracking.

Several variables, besides the presence of water and, in case, of carbon dioxide dissolved in it, may affect the phenomenon of self-healing, such as:

- the mix constituents: Dhir et al. [10] found that self-healing is higher in mortars with a higher con-tent of cement;
- the stress state along the cracks and the steadiness of the cracked state [11];
- the temperature of the water: Reinhardt and Joos [12] found that a higher temperature of water favors self-healing;
- or the alternation between water saturated conditions and exposure to air with different relative humidity, which reduced “the strength developed by a marked degree” [2].

If the mechanisms of self-healing have been quite well understood and reaction products thoroughly characterized from a chemical point of view, the quantitative assessment of its effects on the engineering properties of concrete and cement based materials still needs and deserves a much deeper and more comprehensive dedicated investigation. Most of the surveyed studies [7,13-15] focused on the variation of water permeability and only very few among them [2,10] analyzed the effects on recovery of mechanical properties. Furthermore, most studies only investigated recovery in signal transmission, which, as reported by Aldea et al. [15], was not as spectacular as that in permeability.

In the very last decade lot of attention and a huge amount of research work have been dedicated to “engineered” self healing, also in the framework of the sustainability framework addressed in the introduction, along three main fields of research: self healing engineered with fiber reinforcement, mineral-producing bacteria and proprietary chemical admixtures.

As for the first topic, the first studies on the capacity of fiber reinforced cementitious composites to undergo recovery of their mechanical properties, upon cracking and exposure to suitable environmental conditions, date back to the early eighties [16,17]. In very recent years, the advent of High Performance Fiber Reinforced Cementitious Composites (HPFRCCs) has given renovated impulse to the research in this field. As a matter of fact HPFRCCs are highly conducive to exhibit self-healing capacity. This is concurrent outcome of the mix composition, characterized by high dosages of cement and cement substitutes and low water/binder ratios, and by the formation of stable multiple tiny cracks be-fore the onset of unstable crack localization. Because of the former, large amounts of anhydrous particles, which feature either cementitious or pozzolanic activity, can be exposed to atmosphere humidity and activate self-healing reactions upon cracking. Furthermore, because of the smaller width of each single crack, even complete resealing may be possible, which is also likely to result into a significant or even complete recovery of strength and strain capacity of the material, as a function of the exposure conditions and preexisting damage/cracking conditions [18]. This opens, e.g., interesting perspectives to the use of HPFRCCs in repairing old or damaged structures. First of all, the repairing material is in fact intrinsically more durable, because of its high compactness and of the crack bridging effects exerted by fibers, which help in controlling crack opening and hence prevent or reduce exposure to environment born aggressive agents. Furthermore, because of the self-healing, the material is able to recover its pristine level of durability and strength, with relevant outcomes, e.g., on the life cycle of the structure, as, *e.g.*, shown by Mihashi et al. [19] with reference to improvement in corrosion resistance due the self healing of the cracks.

The precipitation of calcium carbonate due to the biochemical action of suitable bacteria, such as ureolytic ones which convert urea into ammonium and carbonate [20], has been recently noticed as a possible self healing mechanism and promising results of laboratory studies have been published. As pointed out by Jonkers [20], the following two requirements hold in order to apply this technique to a self-healing concrete:

- the lifetime of the bacteria needs to be long enough, comparable to the service life of the structure;
- the addition of bacteria and/or of the necessary bio-cement precursor compounds must not cause the loss of other properties of the concrete itself.

A further technology to engineer the self healing of cracks in cementitious composites consists in the use of different kinds of, in case proprietary, mineral admixtures, such as aluminosilicate materials and various modified calcium composite materials [21]. The self healing action is in this case mainly due to the swelling and expansion effects and to recrystallization. The supply of water or at least moisture is essential, but “since most infrastructures are exposed to rain or underground water, usually this is an easily satisfiable requirement”. The use of the so-called “crystalline additives” is well known with reference to the reduction of concrete porosity and of water permeability of concrete, and hence to the improvement of the tightness and waterproof properties of structural elements, when required. These additives contain substances which react with cement constituents and form calcium silicate hydrates. The reaction propagates through the concrete mass because of osmosis, Brownian motion and progressive involvement of anhydrous cement particles. The reaction products tend to fill the capillary voids, thus resulting in a system impervious to water and other environment born aggressive substances. The reaction consumes the moisture inside the concrete but can also undergo a delayed activation, whenever the material comes back into contact with water and/or environment moisture: this, as a matter of fact, can happen upon crack formation even at later ages. The effects of this kind of additives on enhancing the self-healing capacity of cracked concrete have not been so far investigated and hence deserve being assessed, in the sight of the aforementioned potential applications.

3 EXPERIMENTAL PROGRAMME

In order to quantify the self-healing capacity of concrete containing crystalline additives and its effects on the recovery of mechanical properties, a methodology has been developed and will be presented in this paper. As a first step, prismatic beam specimens, made with both concrete added or not with the aforementioned additive (the mix-design of both concretes is listed in Table 1), are pre-cracked, up to different crack opening levels, by means of a COD-controlled three point bending set-up. Specimens were then submitted to accelerated temperature and humidity cycles representative of winter exposure conditions for different duration. Finally, three point bending tests were performed on either uncracked or pre-cracked specimens and results, in terms of load-crack opening curves, were compared with those obtained from virgin specimens before any “conditioning”. This allowed recovery to be evaluated in terms of actual crack opening, load and stiffness recovery capacity, and related “self-healing” indices to be defined.

31 beam specimens, 50 mm thick, 500 mm long and 100 wide, were cast with each of the mixed concretes; the specimens, after 72h curing in lab conditions under quilts kept continuously wet, were stored in a moist room at 20°C and 95%RH for 35 days.

Table 1. Mix design of investigated concretes.

Constituent	w/out additive (kg/m^3)	with additive (kg/m^3)
Cement type II 42.5	300	300
Fine aggregate 0-8 mm	975	975
Coarse aggregate 8-16 mm	975	975
Water	165	165
(w/c)	0.55	0.55
Superplasticizer	3	3
Aero-Crystallizing Additive	=	3

Strength development was continuously monitored during this period, through compressive strength tests on companion cube specimens, with the side equal to 100 mm, showing no significant difference between concrete containing or not the crystalline additive (Figure 1).

At the end of the curing period, specimens made with each type of concrete were divided into three groups; specimens of two groups, for each concrete, were pre-cracked, according to the 3-point bending test set-up shown in Figure 2, up to (residual) crack openings equal to about 130 and 270 μm respectively, whereas specimens belonging to the third group were left uncracked. It is worth remarking that the bending tests were performed by controlling the Crack Opening Displacement (COD), measured at the mid-span section by means of a clip-gauge.

Specimens were then put into a climate chamber and subjected to the temperature and humidity cycle sketched in Figure 3. Each six-hour cycle was meant to simulate, in an accelerated way, an average autumn day in northern Italy (in Figure 4 the records of temperature and humidity in November in Milan are shown). Specimens were kept into the climate chamber for four weeks. At the end of the first and second week, one third of each group of specimens (uncracked, pre-cracked at both 130 and 270 μm, and both containing or not the additive) was taken out of the chamber: the specimens were finally subjected to three point bending tests, up to failure, still employing the same set-up shown in Figure 2. Figure 5 shows a synopsis of the complete experimental programme, including air exposure and water immersion conditions, which will be not dealt with in this paper.

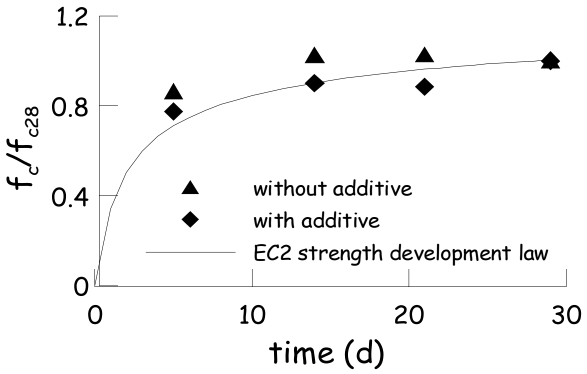


Figure 1: strength development of concrete with and without crystalline additives vs. EC2 provisions.

Figure 2: 3-point bending test set-up

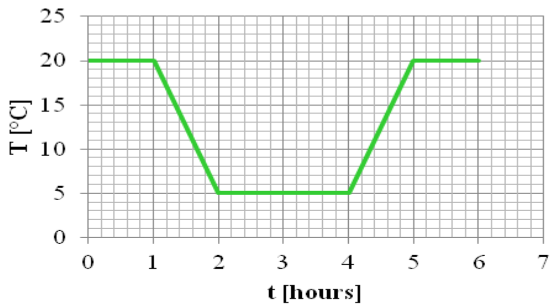


Figure 3: hygrothermal cycles

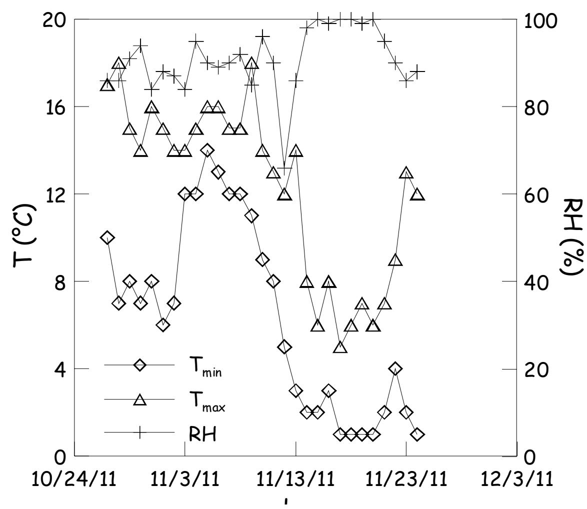


Figure 4: temperature (T) and relative humidity (RH) recorded during November 2011 in Milan

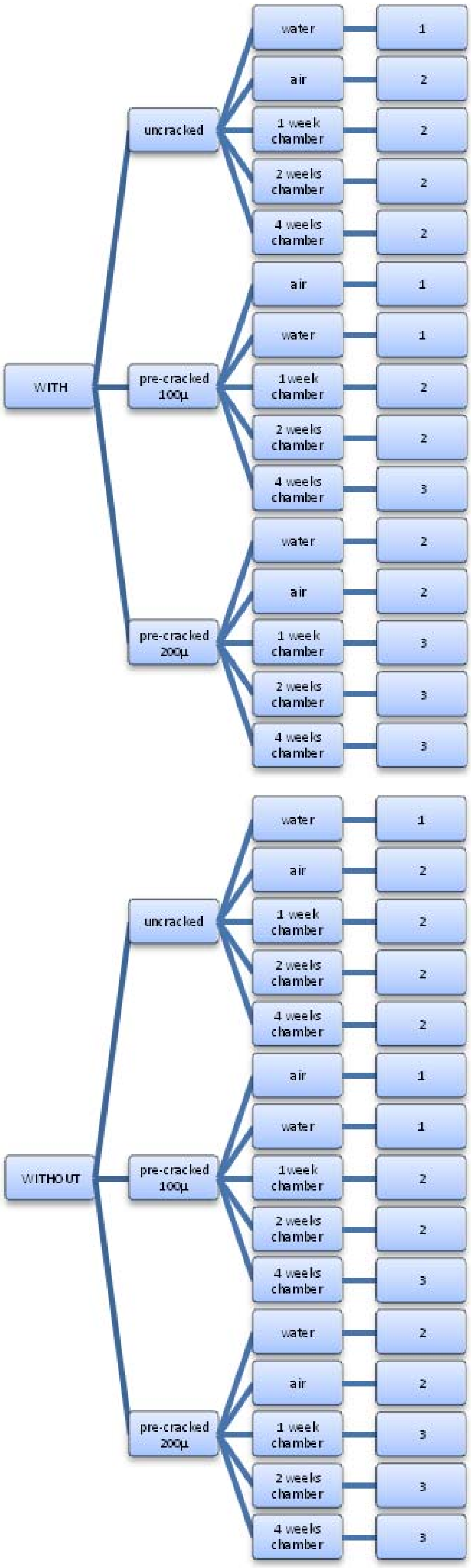
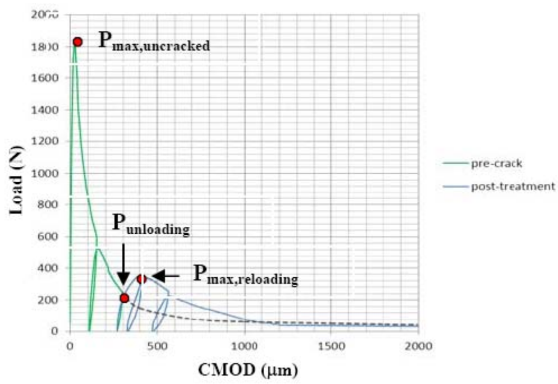


Figure 5:synopsis of experimental program

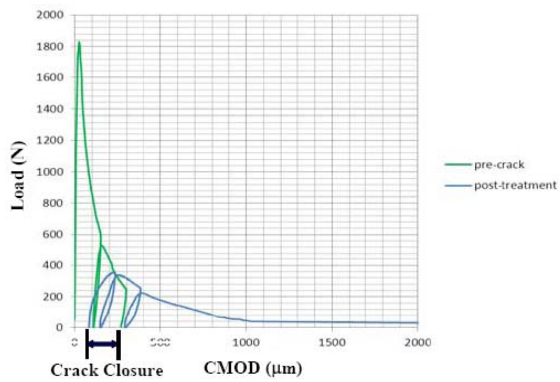
4 EXPERIMENTAL RESULTS

4.1 Evaluation of crack healing

Figure 6a shows an example of Load vs. CMOD curve, as recorded from the same specimen tested in 3-point bending before and after the exposure to hygrothermal cycles in the climate chamber. Consistently with the rationale of the testing program, the curve obtained after the climate chamber exposure has to be interpreted as a reloading of the specimen, following a previous unloading at a prescribed crack opening and the subsequent hygrothermal conditioning. An evident strength recovery capacity has been exhibited by the specimen, which, upon reloading, would have otherwise attained a strength level equal to the one at which is was previously unloaded. It is worth here remarking that all tested specimens featured the aforementioned strength recovery, obviously as a function of presence of the additive, duration of exposure to hygrothermal conditioning and width of the pre-induced crack.



(a)



(b)

Figure 6: example of load-COD curve obtained from 3pb tests on the same specimen before and after T-RH conditioning (strength recovery is evident - a); proposal of a procedure to evaluate crack closure (b)

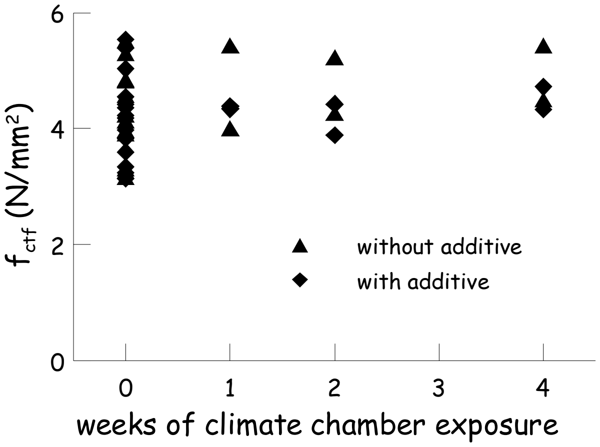


Figure 7: influence of climate chamber exposure on bending strength of unckracked beam specimens

It can be reliably assumed that the strength recovery occurred right because of the self-healing, which led to a partial closure of the previously created crack. The comparison (Figure 7), in terms of flexural strength, between specimens tested at the end of the curing period, i.e. before any kind of exposure or conditioning, and uncracked specimens tested after it, supports this assumption, showing no significant occurrence of continuing hydration for uncracked specimens. On the contrary it can be reliably hypothesized that because of cracking, some un-hydrated material inside the specimens was exposed to environment moisture, which activated, in case also through the aid of the additive, the chemical reactions featuring self-healing.

In order to quantify the crack-strength recovery and the effects on it of the variables recalled above, the following procedure has been adopted. The post-conditioning load-COD curve has been rigidly shifted backward along the horizontal axis (Figure 6b), until its peak load point intersected the softening branch of the virgin load-COD curve. The reasonable matching of softening branches after this shifting may be called to confirm the reliability of the proposed procedure. It is furthermore worth remarking that what has been shown in Figure 6b with reference to one experimental case has been systematically obtained for all the tested specimens. The aforementioned shifting led to a new position the origin of the post-conditioning curve, originally assumed equal to the residual crack-opening upon unloading the virgin specimen after pre-cracking tests. The amount of this shifting can be assumed to quantify the crack closure, and its ratio to the previous crack opening is defined as Index of Crack Self-Healing (ICSH). This index has been plotted, in Figure 8a-b, for concretes both containing or not the additive, as a function of the crack width attained during the pre-cracking bending tests, and of the duration of exposure.

The following statements hold:

- even normal strength concrete, mixed with medium to high water/cement ratios, is likely to exhibit, after conventional aging time (> 28 days), a not negligible crack self-healing; this is most likely due to continuing hydration of anhydrous cement particles present on cracked interfaces and exposed to environment moisture upon cracking. This capacity anyway appears to be randomly scattered and not affected by the duration of the exposure to high relative humidity;
- the addition of crystalline additives enhances the self-healing capacity, which appears to increase with the time of exposure to high moisture and, most of all, is significant even for higher crack openings.

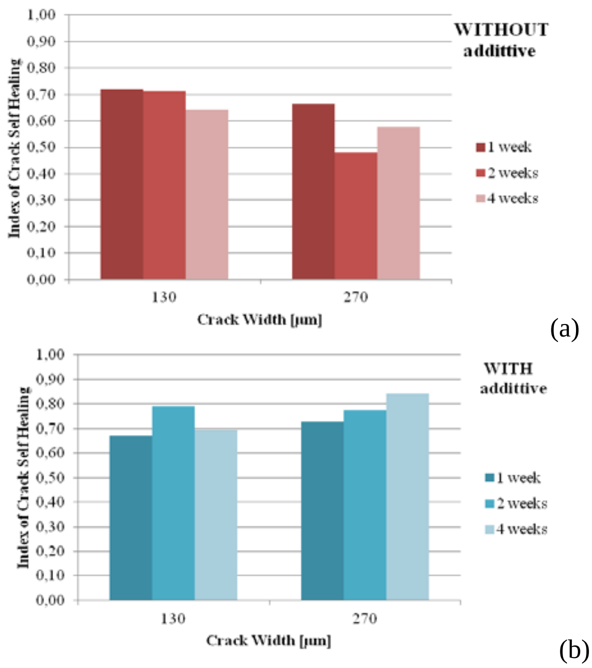


Figure 8: Index of Crack Self-Healing (ICSH), for concretes without (a) or with (b) crystalline admixture, as a function of crack opening and exposure duration.

4.2 Recovery of load bearing capacity

The effects of the crack self healing on the recovery of load bearing capacity have been further investigated. The following two indices of load recovery have been defined, referred to either the material virgin or post-cracking residual strength, (notation in Figure 6a):

$$I_{LR1} = \frac{P_{max,reloading} - P_{unloading}}{P_{unloading}} \quad (1a)$$

$$I_{LR2} = \frac{P_{max,reloading} - P_{unloading}}{P_{max,uncracked}} \quad (1b)$$

In Figure 9 the indices have been plotted vs. the duration of exposure to hygro-thermal conditioning and also as a function of the pre-induced crack width. It can be interestingly observed that the presence of the crystalline additive, acting as a catalyst of the self healing reactions, improves the load recovery capacity as a function of the exposure duration. Furthermore, the larger the crack width, the higher the effects of the self-healing on the load recovery. This can be most likely attributed to the creation of a larger free crack surface and hence to the exposure to environment moisture of larger clusters of un-reacted catalyst and un-hydrated cement particles. On the contrary this tendency has not been measured for specimens made with concrete which does not contain the catalyst additive. As a matter of fact they feature an always lower load recovery capacity, and furthermore scantly sensitive to the exposure duration and negatively affected by the crack width (in this case the larger the crack width the lower the load recovery capacity).

Results appear furthermore to be consistent with the previously estimated Index of Crack Self Healing (ICSH): in Figure 10 the latter has been assumed as the variable governing the load recovery capacity and the consistency and significance of its definition clearly appears, right when referred to the recovery of stress bearing capacity, triggered by the self healing reactions. The improvements, even remarkable, which can be achieved due to the addition of the crystalline additive are evident.

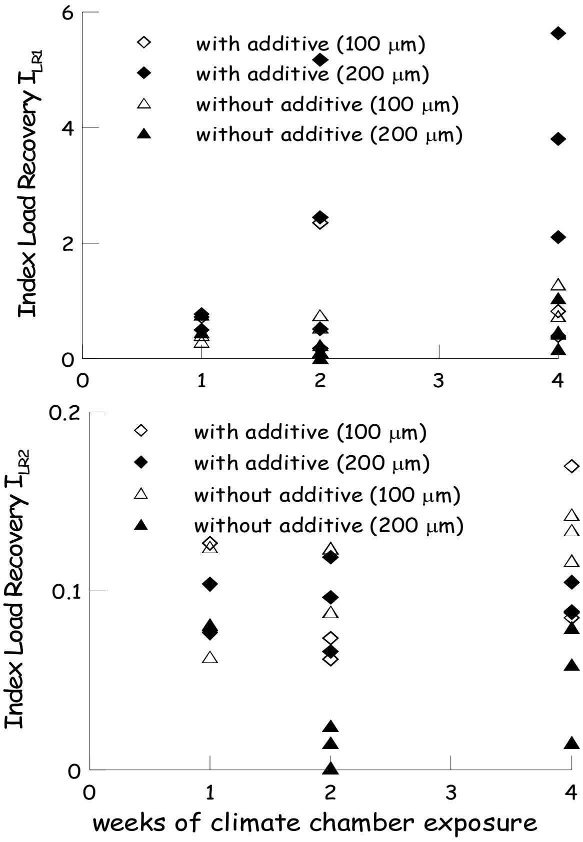


Figure 9: Indices of Load Recovery as a function of exposure duration and crack opening.

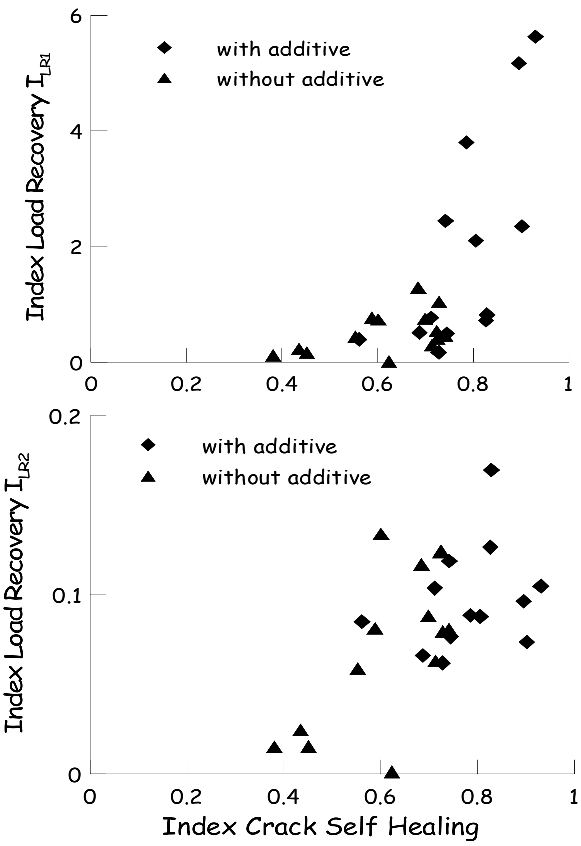


Figure 10: Indices of Load Recovery vs. ICSH.

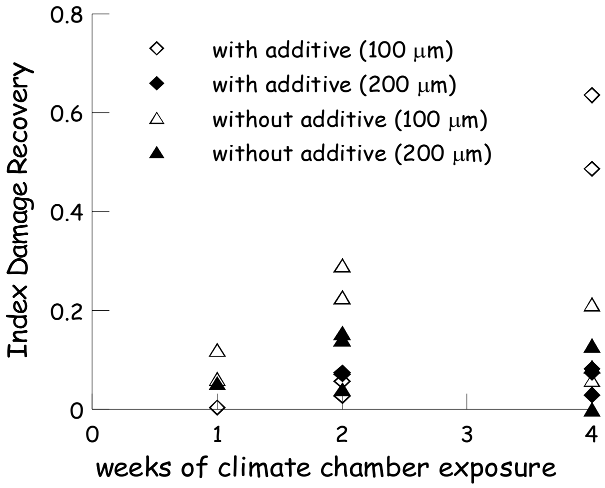


Figure 11: Index of Damage Recovery as a function of exposure duration and crack opening.

4.3 Recovery of stiffness

Evaluation of the effects of the self healing capacity on the recovery of mechanical properties of the material has further gone forward by considering the structural stiffness of the specimens, as estimated from the load-crack opening curves. Because the variation of stiffness can be directly correlated to a damage index, the following Index of Damage Recovery (IDR) has been defined:

$$I_{DR} = \frac{K_{reloading} - K_{unloading}}{K_{initial}} \quad (2)$$

where K denotes the specimen crack-opening stiffness. In Figure 11 the Index of Damage Recovery has been plotted as a function of the duration of exposure to hygro-thermal conditioning cycles and of the pre-induced opening of the crack.

The trend of this index is less immediate than the one detected for the load recovery indices. It can be observed that while for the first two weeks of exposure the concrete without additive performs better than the other one, for longer exposure the performance of the concrete containing the crystalline additive becomes significantly better, whereas the former ones tend to stabilize. It furthermore appears that the recovery of stiffness, and hence of damage, is more significant for smaller crack openings, most likely because of the softer nature of the self healing reaction products.

5 CONCLUSIONS

In this study a methodology has been proposed to measure and quantify the effects of self-healing on the mechanical properties of cement based materials. The methodology is based on pre-cracking beam specimens to prescribed crack-widths, exposing them to suitable real or artificial environment conditions, and, after that, testing them again until failure according to the same set-up employed for pre-cracking. Self-healing capacity has been evaluated by seeking suitable matching between the load-crack opening curves obtained for the virgin specimen and for the conditioned one.

In this paper only artificial exposure conditions, corresponding to autumn Northern Italy climate, have been considered, as a start-up of a more comprehensive research program.

It has been shown that cementitious materials inherently possess, within an acceptable range, some self-healing capacity, most likely due to continuing hydration favored by suitable environment conditions, which is anyway randomly scattered.

The inclusion in the concrete mix of aero-crystallizing admixtures not only enhances the aforementioned self-healing capacity, even up to more than 80% recovery of the crack opening, but also makes it more reliable and consistent.

The proposed methodology, as well as the previous statements referring to the effects of self-healing, needs to be assessed and confirmed with reference to a much wider variability of natural and artificial exposure conditions (different hygrothermal cycles, natural exposure, water immersion, wet-and-dry cycles even in marine-like environment etc.). Characterization of self-healed cracked interfaces through microscopy observation is also needed and currently ongoing. This will be surely instrumental to gain a stronger confidence in the self-healing phenomenon and its effects on mechanical properties of cementitious composites, which is of the utmost importance in order to consistently take them into account in the framework of durability-based design approaches.

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