MICROSTRUCTURAL ANALYSIS OF CRYSTALLINE TECHNOLOGY BY NANOSEM - FEI NOVA 200

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1 General Introduction

Nowadays in building industry is concrete, thanks its technological features one of the most exploited material. It is relatively very stable material with high lifetime, but if concrete structure is not properly protected, happens then with time, that concrete surface layer is degradated. To prevent that are today more and more used backfillings, coatings, sprays and additives on so called "secondary crystallization bases" or "crystalline technology".[1]

2 Functional principle

Crystalline products are dry powder compounds composed of portland cement, silica sand and many active, proprietary chemicals. To create its crystalline waterproofing effect, crystalline products must become an integral part of the concrete mass. It does so by taking advantage of the natural and inherent characteristics of concrete, capillarity and chemical in nature. By means of diffusion, the reactive chemicals in crystalline products use water as a migrating medium to enter and travel through the capillary tracts in the concrete. Basically its main functional principle is that crystalline product chemically reacts with moisture and the natural chemical by-products of cement hydration (calcium hydroxide, mineral salts, mineral oxides and unhydrated and partially hydrated cement particles).[2]

According to contemporary piece of knowledge is concerned that whole cumulated process is accompanied by production of thobermorith gel $3Ca{\cdot}2SiO_2{\cdot}3H_2O$ together with formation of hydratetracalcium aluminate $3CaO.Al_2O_3.Ca(OH)_2{\cdot}12H_2O$. Simultaneously during this fluatation process is proceeds with the rise, in water very insoluble, CaF_2.

At the end as the result of this crystallization, non-soluble crystalline structure, that plugs capillary active pores and tracts of the concrete, is created (average 10^{-7} to 10^{-4}m). Capillary system is no longer accessible for water and moisture from outer environment. Scheme of functional principle can be seen on Fig. 1.

3 Idea

In chapter above has been described how this artificial hydro isolation helps protect concrete against corrosion effect. It is clear, that in this case growth of crystals is main driving issue for full concrete protection. In Xypex Concentrate manual is described that: "Loading of protected concrete surface by pressure water can be done after minimum of 12 days from application (in case of aggressive medium in minimum 18 days)". That gives the idea of minimum time needed for concrete protection. Because crystalline technology has the ability of crystal growth as long as sufficient amount of water is provided, the main idea of experiment was to determinate how crystals develop in pore system for minimum of 12 days as is described in Xypex Concentrate manual.[3]

4 Testing

4.1 Introduction

By the 12 days period for concrete protection has been set the limit for experiment. For comparation of crystal growth has been(based on previous research) created modification based on Xypex Concentrate, working named as

N. N modification is double based composition on cement-polymer platform, where cement matrix was partly replaced by fly ash.[4] Due to analysis can be also seen effect of crystal growth in "environmental friendlier" modification N compared to commercially produced Xypex Concentrate.

Still is main focus of observation has been on main issue - how secondary crystallization is developing in concrete pore system during 12 days period. Is it enough for Xypex Concentrate waterproofing ability?

4.2 Equipment

Analysis has been made by using SEM microscope Nova NanoSEM 200 with module EDAX - Pegasus X4M (EDS/EBSD) in SEMAT laboratories at University of Minho in Portugal. During micro structural observation has been made also EDAX quantification element analysis to determinate evolution of elements in concrete pore system during the test. Sample of crystal developing can be seen in part 4.4 Results.[5]

4.3 Samples and testing

For testing were prepared 3 concrete samples from modification N and Xypex Concentrate. Sample dimension was set to 100x100x100mm. Samples were after 28 days fully laid into the water for 24 hours period. After that, have been from one side treated by modifications and laid back into the water to ½ high of its side length. Treated side was kept above the water level. That procedure guaranteed sufficient water amount for crystallization. In period of 2, 6 and 12 days samples were taken out of the water and prepared for SEM analysis and tested. Samples were taken in depth of 1-1,5cm from the surface protected by crystallization masses.

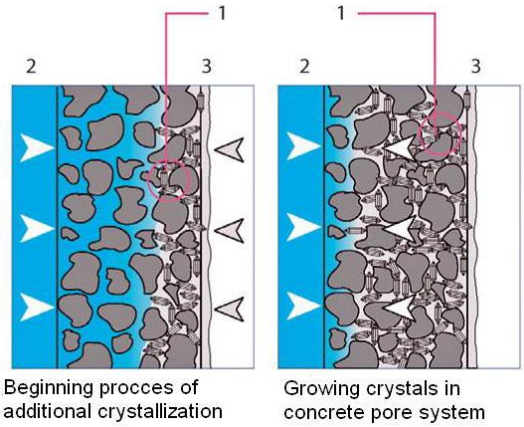

Fig. 1: Schema of functional principle of crystallization backfilling on concrete construction 1 – plugging crystals, 2 – surface loaded by water pressure, 3 – surface with crystallization coating[3]

4.4 Results

In this chapter are presented representative figures taken from samples tested during observation process. Figures represent main issue discovered during several observations. Figures have been modified (cutted from original picture) to focus on main issue.

4.4.1 2. Days after beginning of crystallization

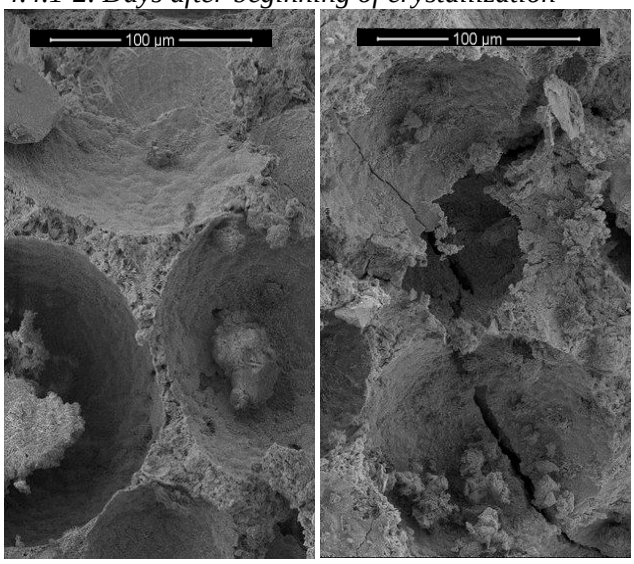

Fig. 2: Xypex Concentrate magnification 1000x (left figure Xypex Concentrate, right figure modification N)

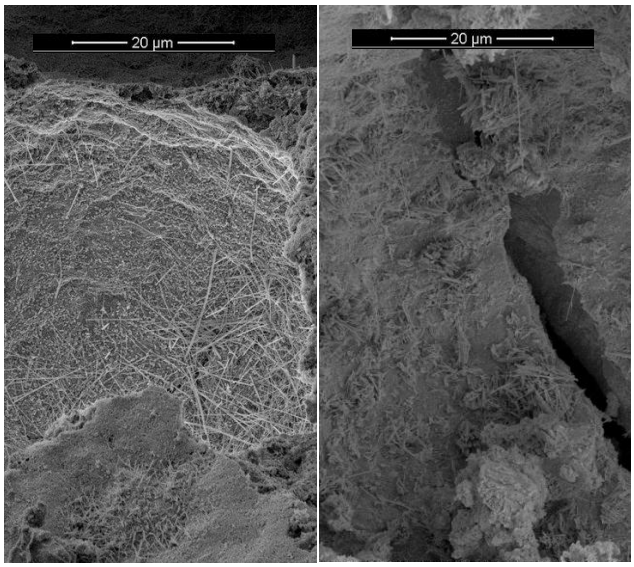

Fig. 3: Xypex Concentrate magnification 4000x (left figure Xypex Concentrate, right figure modification N)

4.4.2 6. Days after beginning of crystallization

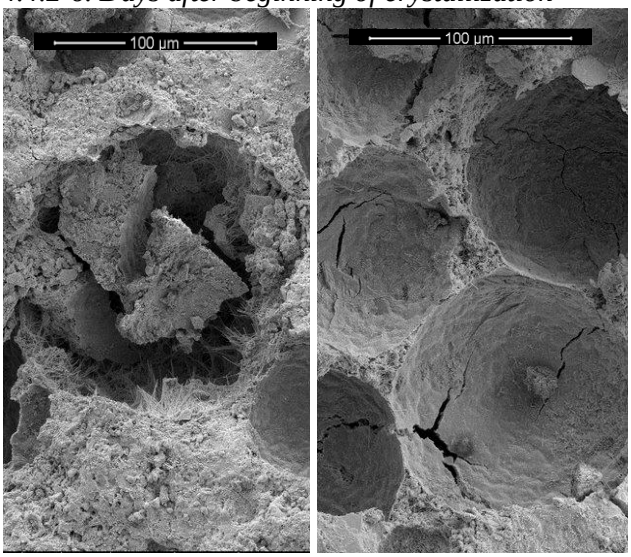

Fig. 4: Xypex Concentrate magnification 1000x (left figure Xypex Concentrate, right figure modification N)

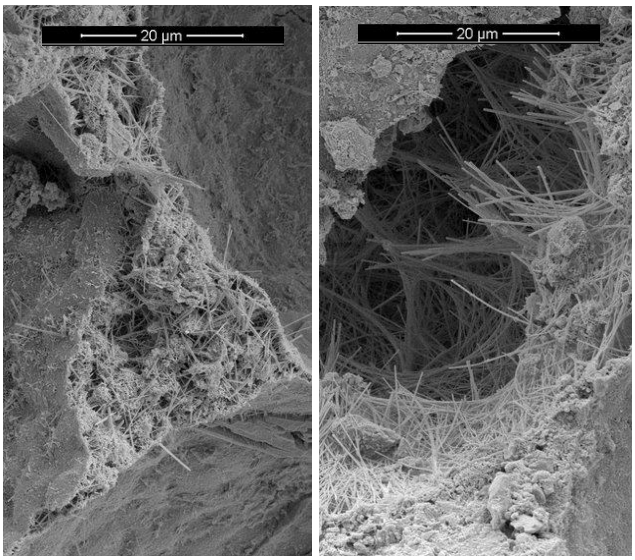

Fig. 5: Xypex Concentrate magnification 4000x (left figure Xypex Concentrate, right figure modification N)

4.4.3 12. Days after beginning of crystallization

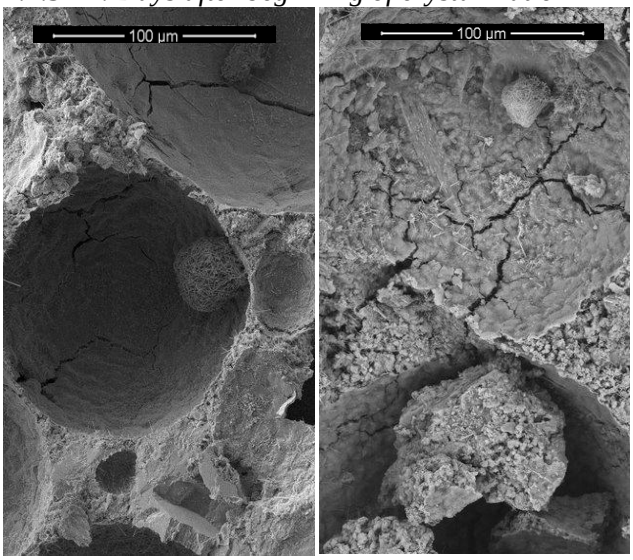

Fig. 6: Xypex Concentrate magnification 1000x (left figure Xypex Concentrate, right figure modification N)

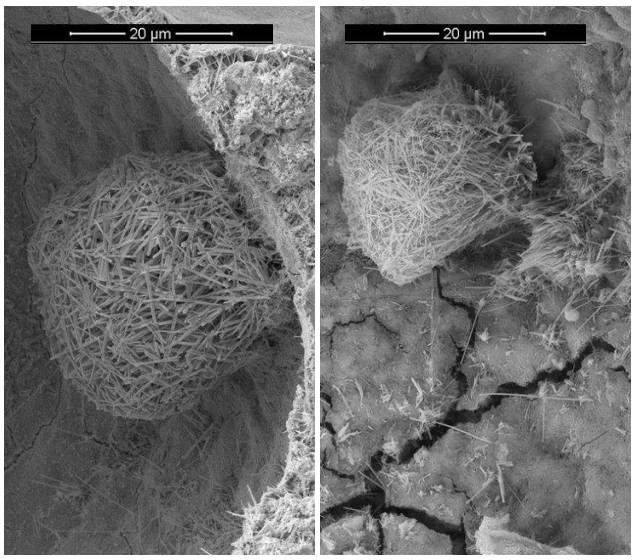

Fig. 7: Xypex Concentrate magnification 4000x (left figure Xypex Concentrate, right figure modification N)

4.5 Conclusion

SEM analysis has proofed growth of crystalline structure in concrete pore system. During 12 days after application needle shaped crystals has been developed and with continuous time its size and structure has been more and more complex. During observation was really clear, that crystal development is strongly depended on the presence of water. In places with no water access even after 12 days did not grew any crystals. Was also observed difference between Xypex and coating "N modification" in amount of crystals and its complexity (see part 4.4 above). During analysis of Xypex Concentrate was very often observed ball shaped formation which filled only parts of the pores. This could cause presence of limited amount of water (perhaps only"single drops" of water). Based on observation can be conclude, that crystalline technology has the ability of growing crystals in couple of days and its effectiveness is strongly dependable on the water presence in pore system.

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