

Investigation of Crystalline Products in Cored Concrete Sample

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1. Introduction & Objective of The Investigation

Three cored concrete sample were obtained from the abutment of Breidbalakvisl Bridge in Iceland. The abutment was treated with Xypex Concentrate in 1993. The core samples are named "1", "2", and "3". Each core was cut into slices. The portion of each core in the distance within 8 mm from the surface is denoted by "A" (namely 1A, 2A, 3A) while the portion in the distance between 150 – 165 mm is denoted by "E" (namely 1E, 2E, 3E). These six slices were selected for the investigation. **Figure 1** shows the samples as received.

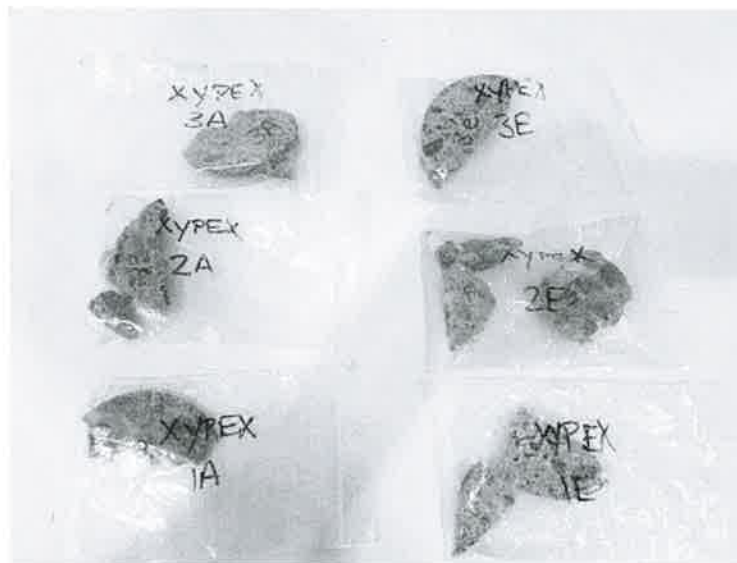


Figure 1: As-received samples for crystal investigation.



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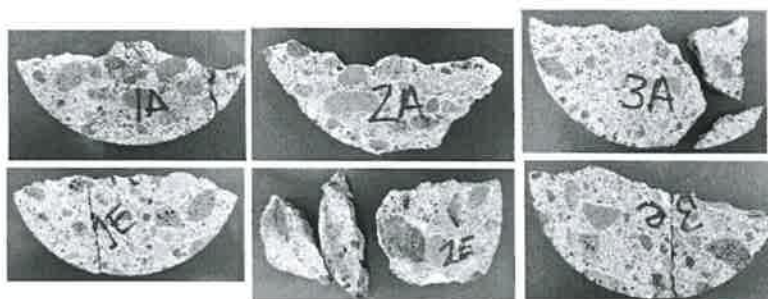


The main objective of this investigation work is to investigate the crystalline products formed in each samples and compare them. This is performed by the use of Scanning Electron Microscope (SEM). The samples were prepared and investigated as described in the following sections.

2. Sample Preparation for Crystalline Investigation

The surface slices (1A, 2A, and 3A as shown in Figure 2a) and three slices from deeper portion of each core 1E, 2E, and 3E (as shown in Figure 2a) were broken into smaller pieces with an approximate size of 5 mm.

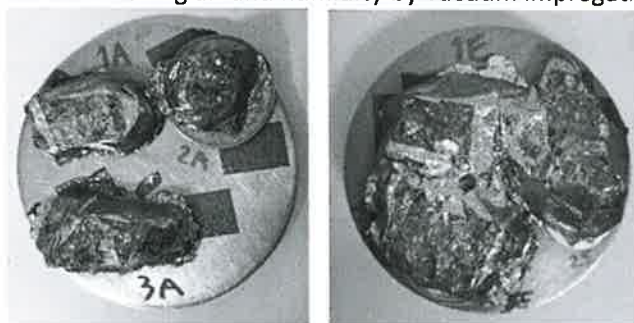
Two small pieces were selected from each slice to be investigated for presence of crystalline products. These small pieces of samples were then kept in vacuum chamber with silica gel (as shown in Figure 2b) for 3 days in order to dry the sample. The sample were then coated by gold spraying (as shown in Figure 2c) just before performing the investigation using SEM.



(a) Six different core samples



(b) Drying of specimens using by silica gel and removing air and humidity by vacuum impregnation



(c) Specimens after gold coating

Figure 2: Sample preparation procedures for investigation using SEM


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3. Investigation Procedure

The coated sample were inserted into the SEM/EDX machine (JEOL JSM-6610LV) shown in Figure 3. Surface morphology of each sample were then observed thoroughly. The SEM photos were then taken at five different locations in each sample in order to represent the overall morphology found in each sample. At each location, photos with three different magnification levels (200x, 1000x, 5000x) were taken for references.



Figure 3: SEM/EDX machine used in this investigation

Surface morphology observation is performed based on Secondary Electron Image (SEI) mode. In this case, the SEM photo with 200x magnification shows the air voids smaller than 100 μm being filled with some crystals which are not common in ordinary Portland cement concrete. The SEM photo with 1,000x magnification shows that the crystals have different shapes. Although most of the crystals are needle-like crystals, some with plate-like shape were also found. It can be also observed that the length of needle-like crystal is in the range of 10-40 μm while the size of plate-like crystal is approximately 5-10 μm . The photos with 5,000x magnification shown in Figure 4(c) provide additional details of the size of needle-like crystals. The diameter of the needle-like crystal is around 0.5 μm .

Similar investigations were performed for all samples both from the near-surface region (1A, 2A, and 3A) and from the deeper location (1E, 2E, 3E). The full set of SEM images is provided in the Appendix. The overall evaluation for the crystalline products in each sample is reported in the next section.

4. Investigation Results

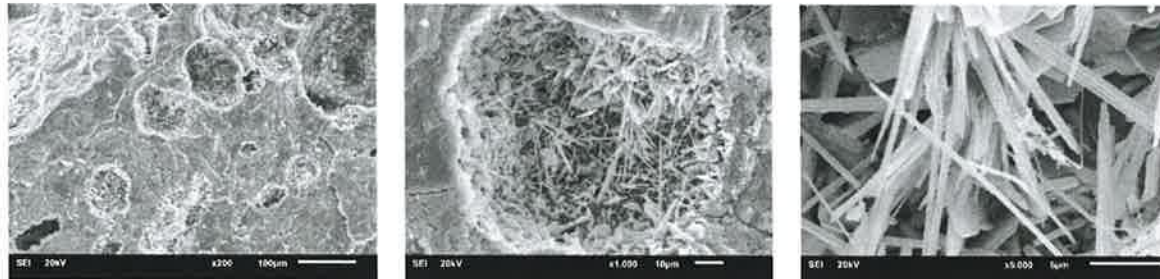
The examples of SEM photos (Sample 1A-1, 1A-2, 3A-1, 3E-1) are shown in **Figure 4**, **Figure 5**, **Figure 6**, and **Figure 7**.

Table 1 summarizes and compares characteristics of crystal formation found in different samples. *In all samples, special types of crystals are found.* These crystals are not common in the Portland cement concrete. However, the intensity of crystal formation as well as the shape of the crystal varies

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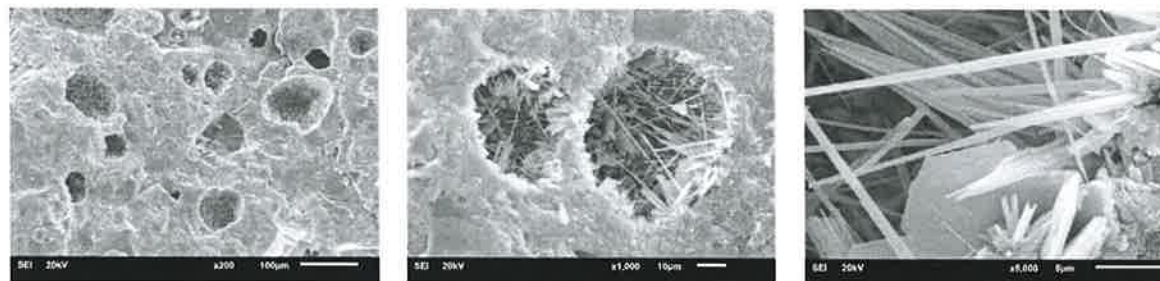
considerably. Two types of the most commonly found crystals are the needle-like crystals and the hexagonal plate-like crystals. However, there are also some different shape of crystal found in some samples and, in some sample, the crystal seems not to be fully developed although some formation could be observed on the surface of voids.



(a) 200x magnification

(b) 1,000x magnification

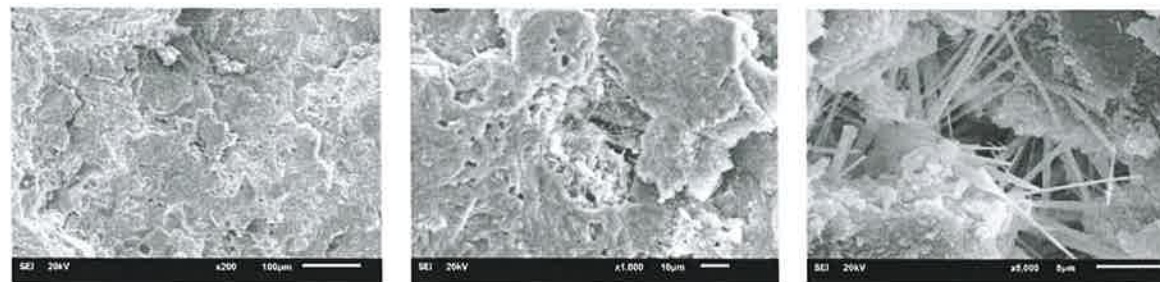
(c) 5,000x magnification

Figure 4: Example of SEM photos (Sample 1A -1 at the 3rd location)

(a) 200x magnification

(b) 1,000x magnification

(c) 5,000x magnification

Figure 5: Example of SEM photos (Sample 1A -2 at the 5th location)

(a) 200x magnification

(b) 1,000x magnification

(c) 5,000x magnification

Figure 6: Example of SEM photos (Sample 3A -1 at the 4th location)

(a) 200x magnification

(b) 1,000x magnification

(c) 5,000x magnification

Figure 7: Example of SEM photos (Sample 3E -1 at the 3rd location)


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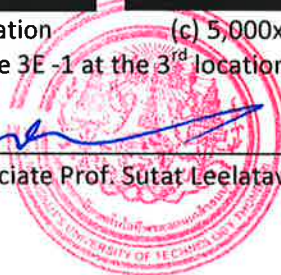


Table 1: Characteristics of crystals found in each sample

Core ID	Near-Surface Sample (A) (within 8 mm from surface)		Sample from Deeper Portion (E) (approximately 150 mm from surface)	
1	1A-1	A lot of crystal found. Most (90%) of the crystal is needle-like with few crystal in the different forms	1E-1	There is noticeable needle-like crystal but the formation intensity is much lower than "1A-1". However, existence of small crystal is extensively noticeable on surface.
	1A-2	A lot of hexagonal plate-like crystals with few needle-like crystal.	1E-2	Not so much crystal found throughout the investigation on this sample.
2	2A-1	A lot of crystal found. The characteristics and shapes of crystal also vary a lot.	2E-1	Not so many clear crystal is observable. However, some products obviously formed on the surface of voids.
	2A-2	A lot of crystal found. Most of voids are filled with crystals. However, it was observed that, in some voids, the crystal could not fully developed.	2E-2	Crystal extensively forms on the surface of air bubble. However, shapes and characteristics of the crystal varies a lot. Many shape of small crystal found
3	3A-1	Not so much large crystal found although many small hexagonal crystals could be observed at the surface of air bubble. Some small size air voids are filled with needle-like crystals.	3E-1	Considerable amount of needle-like crystal or hexagonal plate-like crystal found. However, it seems that the crystal could not fully develop in some voids. Denser crystal development is found in smaller voids. (More crystal found than in 1E-1)
	3A-2	Not so much large crystal found. Some substance is observed on the surface of voids but the shape of crystal is unclear.	3E-2	Very strange shape of crystal is found. The crystal seems to be distributed well on the surface of void. However, the amount of crystal is not so much.

It is important to note that this report is based on taking photos from five locations in each sample. Although the location of photo shootings was carefully selected to be a good representative of overall characteristics found in each sample, they may not perfectly represent the real condition of the samples. The information presented here may be suitable only for qualitative comparison and should not therefore be used for quantitative judgment.

The high variation of the crystal found in this investigation should be also emphasized. For near-surface samples, the sample "1A" show approximately 90% of needle-like crystal with the rest as plate-like hexagonal crystal. The sample "2A" show several forms of crystals including needles, sheet, and sphere, etc. While, the sample "3A" show plenty of small hexagonal crystals and sphere products with very little of needle-like crystal. Similarly high variation of crystal is found in the case of samples from deeper portion (1E, 2E, and 3E).

Also, it should be noted that, since only surface morphology is investigated by using SEM, no information about chemical composition of each type of crystal was investigated.


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5. Conclusions

- In all samples, a certain amount of crystal formation could be observed. The type and amount of crystal found is not usual crystal found in Portland cement based system.
- The amount of crystal formation as well as the shape of crystal is highly variable.
- A higher density of crystals was found in the near surface samples. Crystals were also found in the deep samples but to a lesser extent.



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