PHYSICAL INFRASTRUCTURE CENTRE
RESEARCH, INVESTIGATION & DEVELOPMENT

TEST REPORT No: CET [redacted]

TEST REPORT DATE: 04 August 1998

CLIENT: [redacted]
Brisbane
QLD 4000

REFERENCE: [redacted]

TEST DATE: May - July 1998

TEST DESCRIPTION: Comparison between cracked surfaces on treated and untreated concrete specimens.

TEST SPECIFICATION: As requested by the clients Engineer[1] [redacted]

TEST EQUIPMENT: JEOL 840A SEM Microprobe.

SAMPLE DATA: QUT staff fabricated four (4) concrete beams 1000 x 300 x100mm thick, using standard 32 MPa. concrete as supplied by Pioneer Concrete (Qld) Pty Ltd. (Docket No.0138139).F42 fabric having 20mm cover from top and bottom face was used in the fabrication. Each beam was given a brushed surface on the top face.

TEST PROCEDURE: When the surface became trafficable (approximately 3hrs), two of the beams were given a surface coating of Xypex using a brush application in the proportion of 5 parts powder to 2 parts water at an application rate of $1kg/1.2m^2$.The batch was identified as Xypex No. 0478A. The beams were then covered in hessian sacks and kept moist for 24hrs.

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The beams were then demoulded and cured for a further 6 days in a standard fog room. Full depth cracks were then propagated in the central part of the beams. For each of the treated and untreated beams there was one hairline and one 0.5 - 1mm crack.

The top surfaces of the beams were then dammed to 30mm depth, filled with water and returned to the fog room. After 7 days the beams were removed from the fog room and placed in ambient conditions for a further 14 days with water still being applied to the dammed surface.

A 75mm dia core was then cut through each beam, ensuring the crack was roughly at the diametric of the core. Each core was then immediately placed in a vacuum desiccater for 7 days. The cores were then delivered to our Electron Microscopy facility in a sealed desiccater jar.

OBSERVATIONS

On both the treated and untreated hairline crack specimens the flow of water through the cracks, decreased to a damp line on the bottom face, after 21 days damming. The flow of water through the 0.5 - 1mm cracks on the other specimens showed some decrease after 21 days.

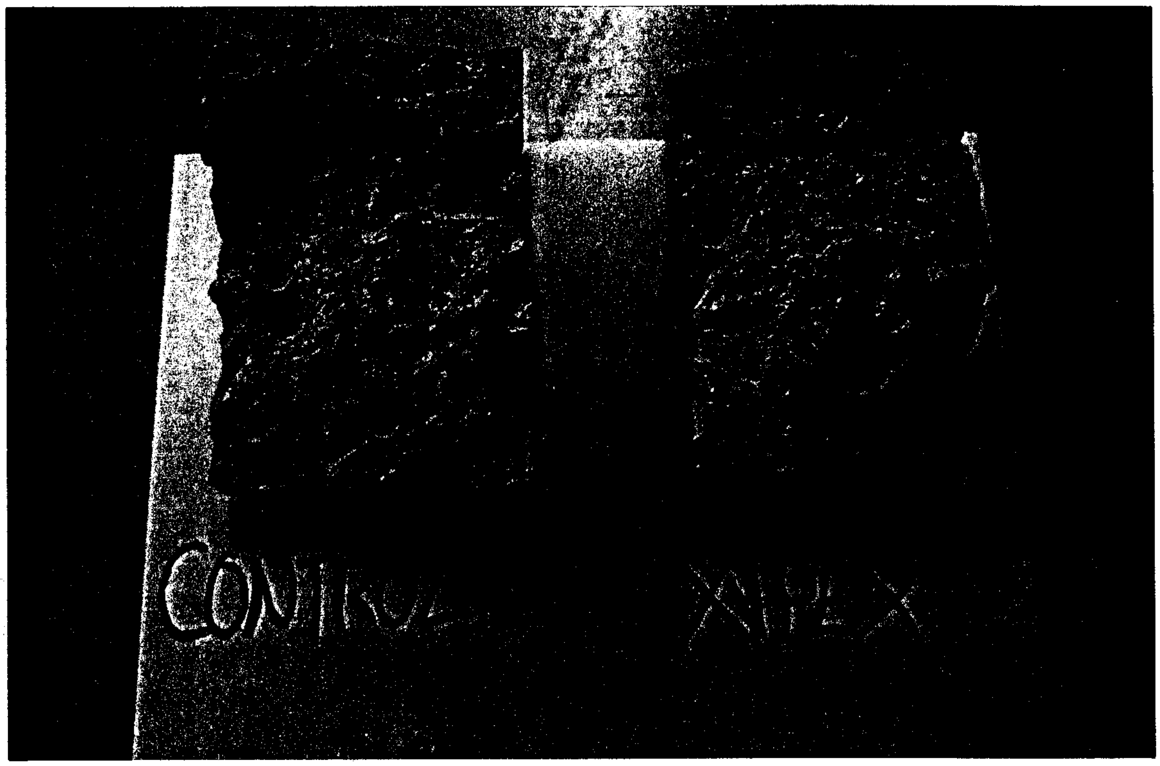
On opening the cracked faces the hairline cracks showed a fine matrix growth and the 0.5 - 1mm cracks showed a heavy matrix growth. Visually (naked eye) there appeared to be no obvious difference between the matrices other than a slight brown tint to the non treated as opposed to the treated surface.

ELECTRON MICROSCOPY

Report and images of the Scanning Electron Microsope are attached.

TESTING OFFICER
C.SMITH

CHECKED BY
D.CORBETT



Details of Cracked Surfaces

Analytical Electron Microscopy Facility
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.29 July 1998

Mr David Corbett
Physical Infrastructure Centre - Research, Investigation and Development
School of Civil Engineering
QUT
Gardens Point, Brisbane, QLD

Re: **Scanning Electron Microscopy and X-Ray Microanalysis of Xypex-Treated Concrete**

Dear Mr Corbett,

Please find enclosed the results of our examination of fracture surfaces of Xypex-treated concrete and untreated concrete (control) using scanning electron microscopy (SEM) imaging and X-ray microanalysis. The SEM shows the crack surface at high magnification, while the microanalysis technique provides information on the elements present in the surface. All elements in the periodic table, except for the light elements below Na (Z=l 1), can be detected with the current equipment.

Pieces were broken off the top of the supplied fractured concrete cores and mounted so that the original fracture face (at right angles to the top surface of the concrete) was uppermost. The pieces were then coated with a thin layer of carbon in a vacuum evaporator to provide electrical conduction in the SEM, and the fracture surfaces viewed at 15kV in a JEOL 840A SEM/Microprobe. The areas examined were within about 15mm of the original top surface of the concrete.

Results

1. No unique element could be detected in the Xypex/cement powder additive that could be used to track the presence of the Xypex. The elements detected were Mg, Al, Si, S, Ca and Fe, all of which are also found in concrete. However, calcium (Ca) was by far the most predominant element in the powder.

2. The fracture surface of the Xypex-treated concrete was covered with a fine deposit composed of crystals roughly 5μm in size (Figure 1). Some deposits exhibited a characteristic polyhedral structure (Figure 3). Microanalysis of the crystals showed only an X-ray peak for Ca (Figure 11), suggesting a compound such as calcium oxide, carbonate or, most likely, hydroxide. Some Ca-rich larger grains on the surface were also found to contain S and Al (Figure 12).

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3. In the treated concrete, small fibrous structures were sometimes observed amongst the crystals (Figure 2), but they were too small to be analysed accurately because of the effect of electron beam spread. These fibres were not seen on the fracture surface of the untreated concrete.

4. The fracture surface of the untreated concrete displayed a more diverse range of grain shapes and sizes than the treated surface, including many more larger crystals and grains (Figure 4). Features such as the multilayered crystal shapes (top of Figure 4) and radial clusters of Ca-rich crystals (Figure 5) were not observed in the treated concrete. However, the small Ca-rich crystals similar to those on the treated surface were also present in the untreated concrete, in association with a range of larger grains (Figure 6). Some of the larger material, including the grain at the centre of the rosette in Figure 5, was found to contain very high levels of Mg (Figure 13). Such Mg-rich constituents were not detected in the treated sample.

5. Overall X-ray microanalyses of a roughly 2x3 mm area of the fracture surfaces gave the X-ray spectra in Figure 9 (treated concrete) and Figure 10 (untreated concrete). X-rays are only produced within a depth of about 5um of the exposed surface, and therefore underlying structures are not being measured by this method. Comparison of the two spectra, and taking into account the differences in peak intensity (indicated by the vertical scales), suggests that the Xypex-treated crack surface had higher levels of Ca and Si, somewhat higher levels of Al and K, and a lower level of Mg, than the untreated surface.

6. Figures 1-6 and the associated spectra are from exposed surfaces of narrow cracks. In a further study, the surface of a larger crack in treated and untreated concrete samples was examined. The surface of the untreated sample is shown at high magnification in Figure 7, and that from the treated concrete in Figure 8. Though there are no marked differences between the surfaces, there is some suggestion in Figure 8 of a smaller grain size and the appearance of some fine lacy or fibrous material (arrows).

7. X-ray spectra from the surfaces of the large cracks are given in Figure 14 (treated sample) and Figure 15 (untreated). Direct comparison of the two spectra, taking into account the differences in vertical scale and peak intensity, suggests that on average the treated crack surface contained more Ca and Mg, but less Fe, than the untreated material. This contrasts with the results for the narrow cracks examined (point 5 above), in which the treated surface showed a reduced average level of Mg. However, in both cases, the average levels of Ca were markedly higher in the treated crack surfaces.

In summary, some differences were observed in the fracture faces of the Xypex-treated and untreated concrete samples that were examined in the SEM. The untreated surface contained a diverse range of grain or crystal sizes and shapes, particularly larger material. The treated surface was mainly covered with small Ca-rich crystals, and larger grains were not as evident. However the Ca-rich crystals were also present to some extent in the untreated sample.

X-ray microanalysis of the fracture surfaces of the narrow and large cracks showed that in both cases, the average level of Ca was higher in the Xypex-treated concrete than in the untreated concrete. Ca was also the major constituent of the Xypex/cement powder additive. The differences in the treated and untreated surfaces with respect to other elements (esp. Si and Mg) were not consistent and may reflect the particular concrete components that happen to be exposed in the analysed surfaces.

Though the crack surfaces of the treated and untreated material do not show marked visual differences, the results suggest the presence of smaller crystals and higher average levels of Ca on the crack surfaces in the Xypex-treated concrete. Thus there may be some modification of secondary crystal formation in the treated material.

Yours faithfully,

Dr Thor Bostrom
Lecturer/Research Scientist

Loc Duong
Technician

Analytical EM Facility, QUT

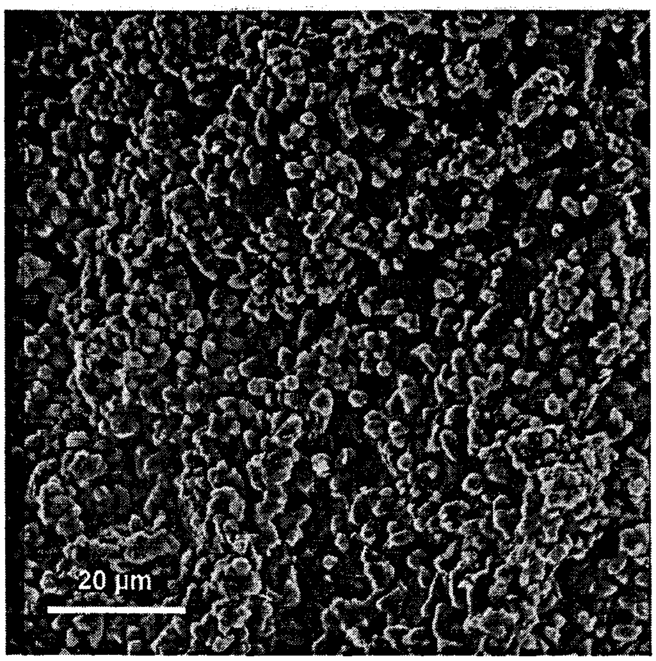

Figure 1 (Treated)
Ca-rich crystals on the fracture surface

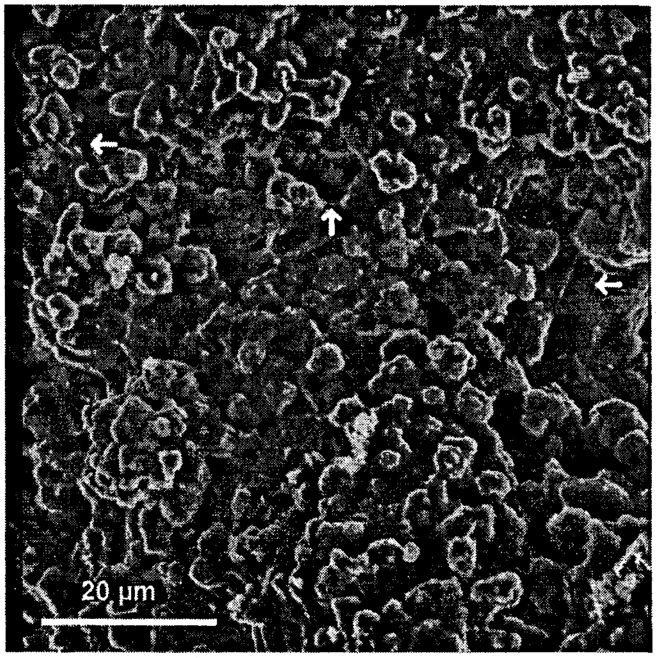

Figure 2: (Treated)
Detail of crystals with occasional fine fibrous material (arrows)

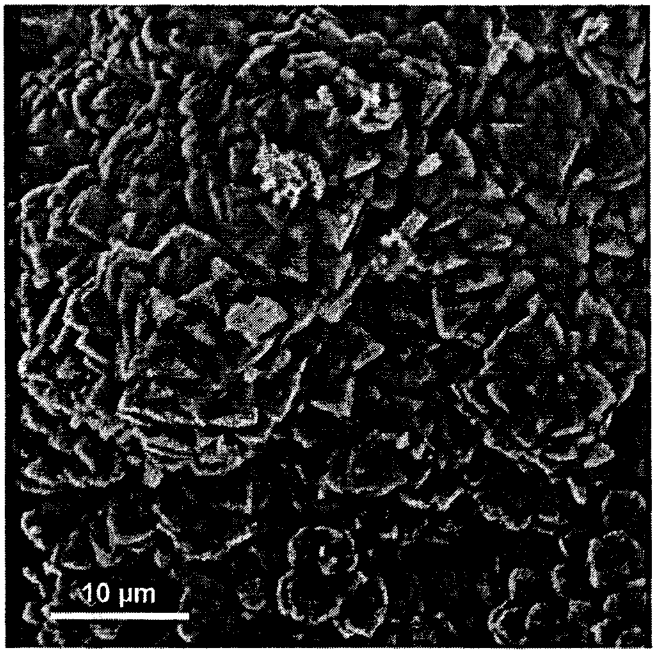

Figure 3: (Treated)
Cluster of characteristic Ca-rich crystals at high magnification

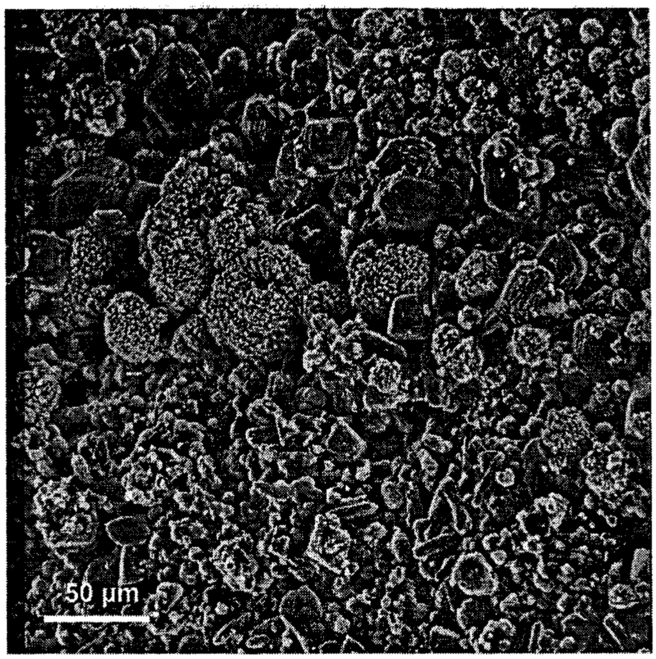

Figure 4 (Untreated)
Fracture surface

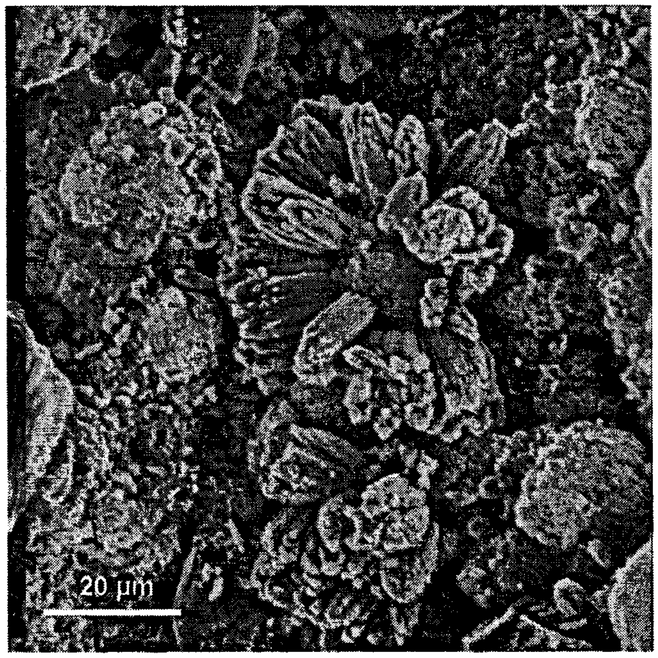

Figure 5 (Untreated)
Detail of crystal growth on fracture surface

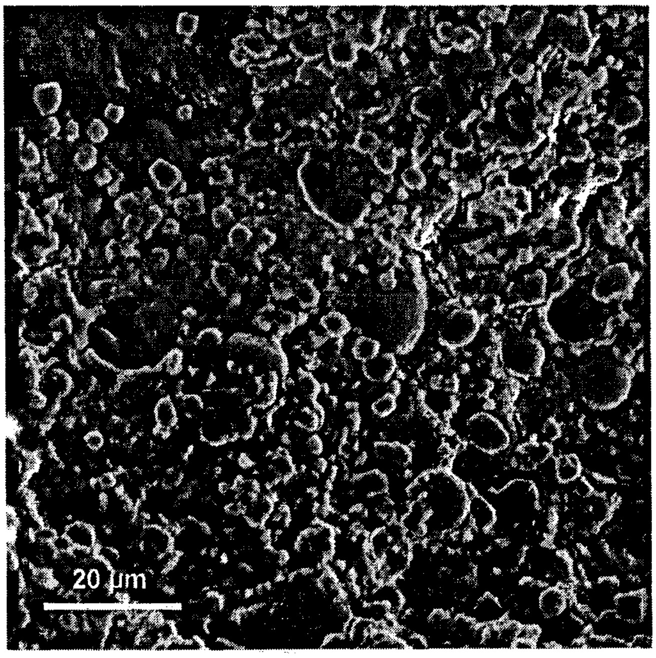

Figure 6 (Untreated)
Small Ca-rich crystals plus larger grains on surface

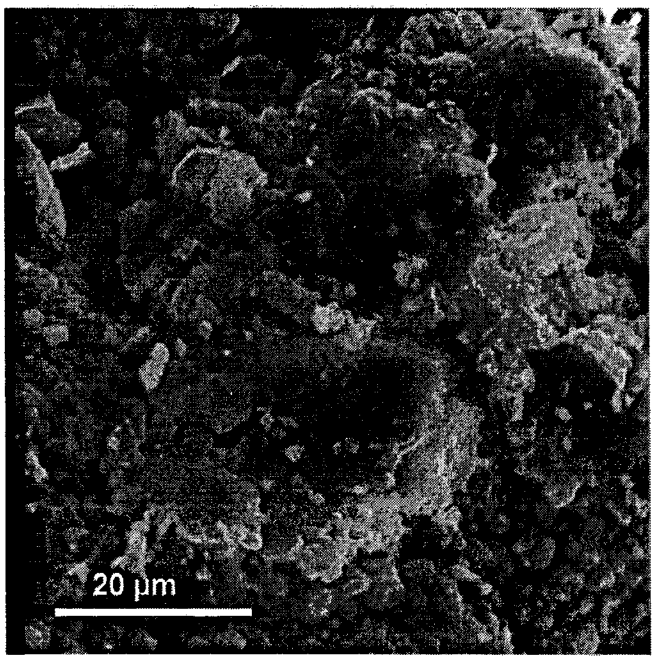

Figure 7 (untreated)
Surface of large crack

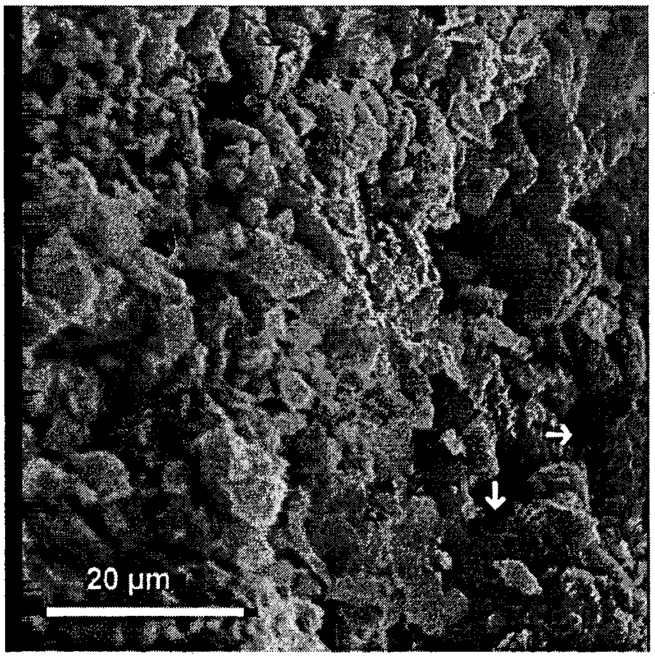

Figure 8 (treated)
Surface of large crack in treated concrete, with some small fibrous material (arrows)

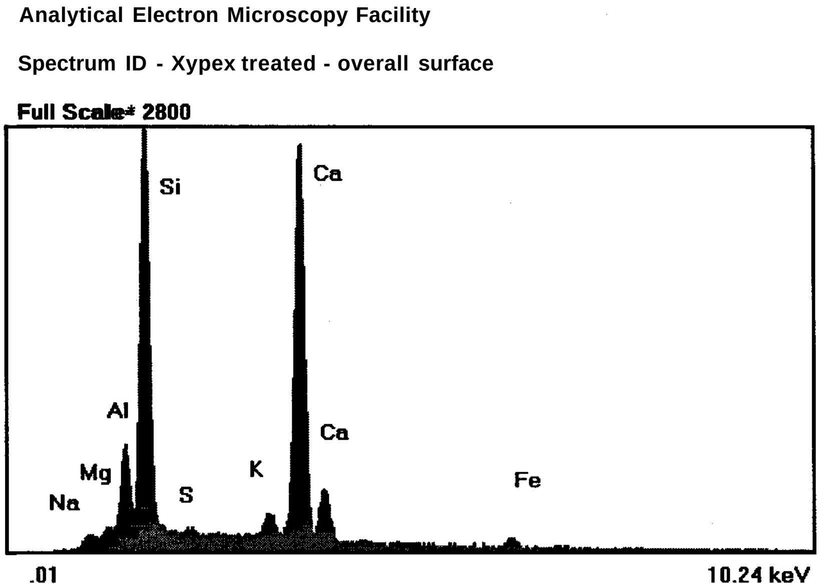

Figure 9 - X-ray spectrum from an overall large area of the fracture surface of the Xypex-treated concrete

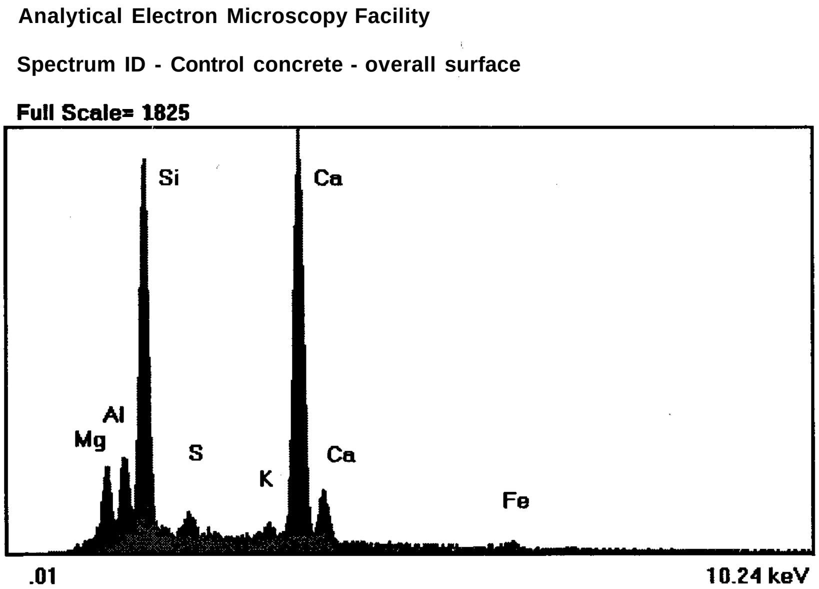

Figure 10 - X-ray spectrum of an overall large area of the fracture surface of the untreated concrete

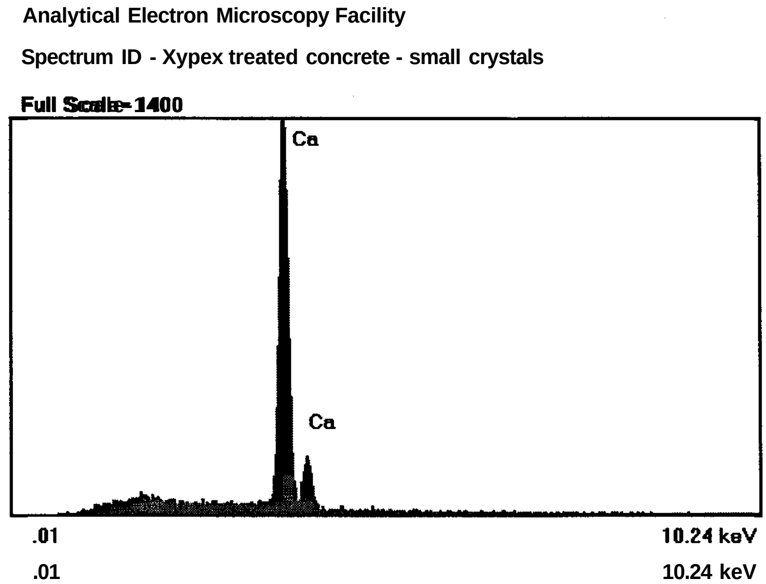

Figure 11: Treated concrete - small crystals on fracture surface

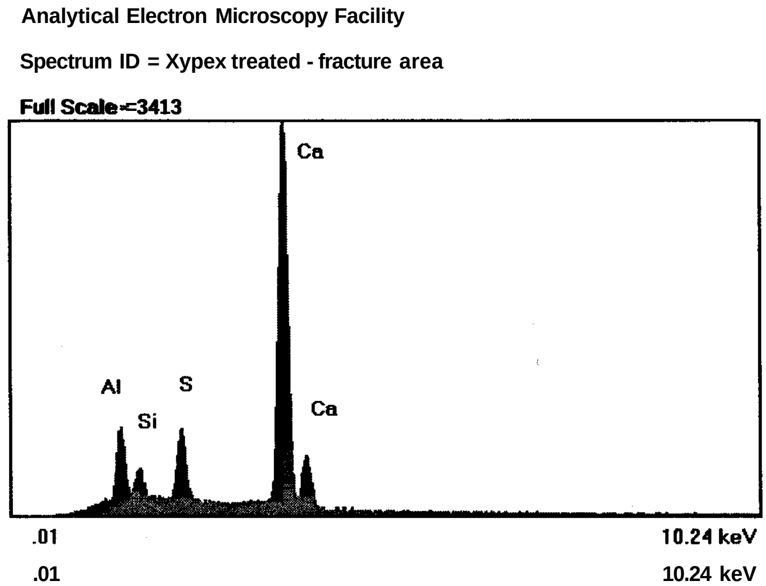

Figure 12 : Treated concrete - surface material with Ca and S

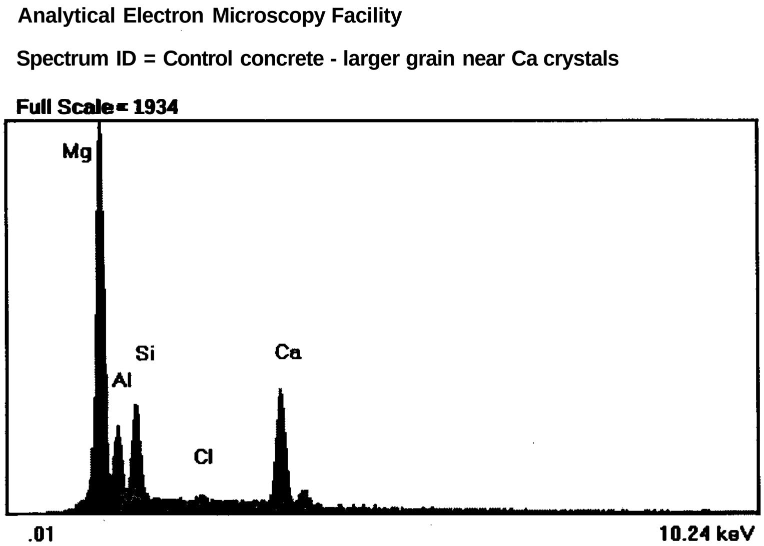

Figure 13 : Untreated concrete - large grains with high levels of Mg

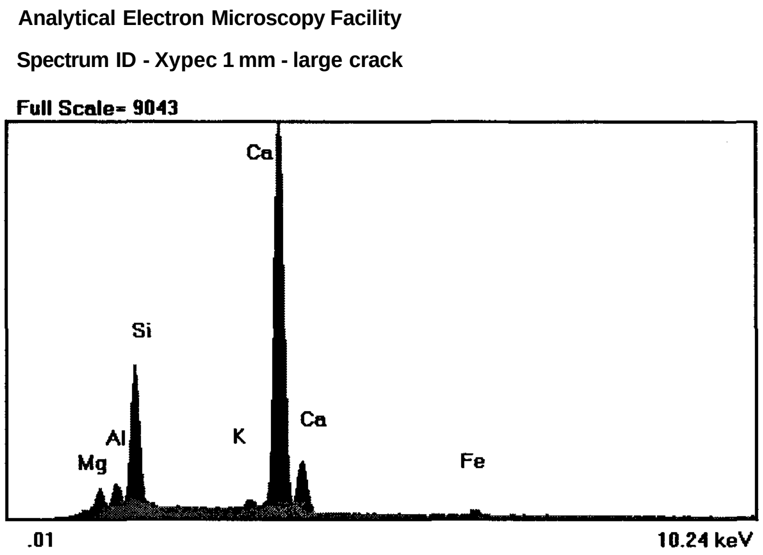

Figure 14 : Surface of large crack (treated concrete)

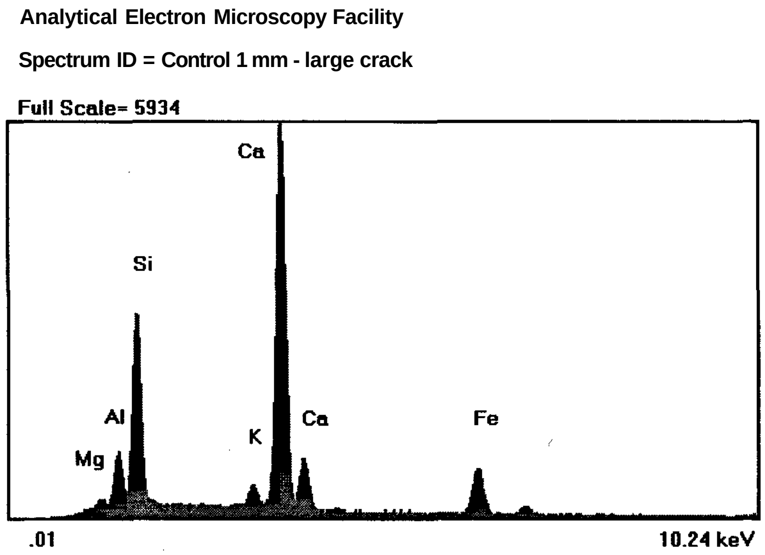

Figure 15 : Surface of large crack (untreated concrete)