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## Getting the small details right!

### Micro analysis tools for concrete examination

### Editorial

We have dedicated this issue of our Concrete News to micro examination of solid materials as concrete, ceramics and metallic materials.

FORCE Technology is using sophisticated optical and scanning electron microscopes (EDS, SEM and FIB/SEM) primarily for investigations of metallic components. Newly these instruments were found useful for the characterization of concrete and similar composite materials.

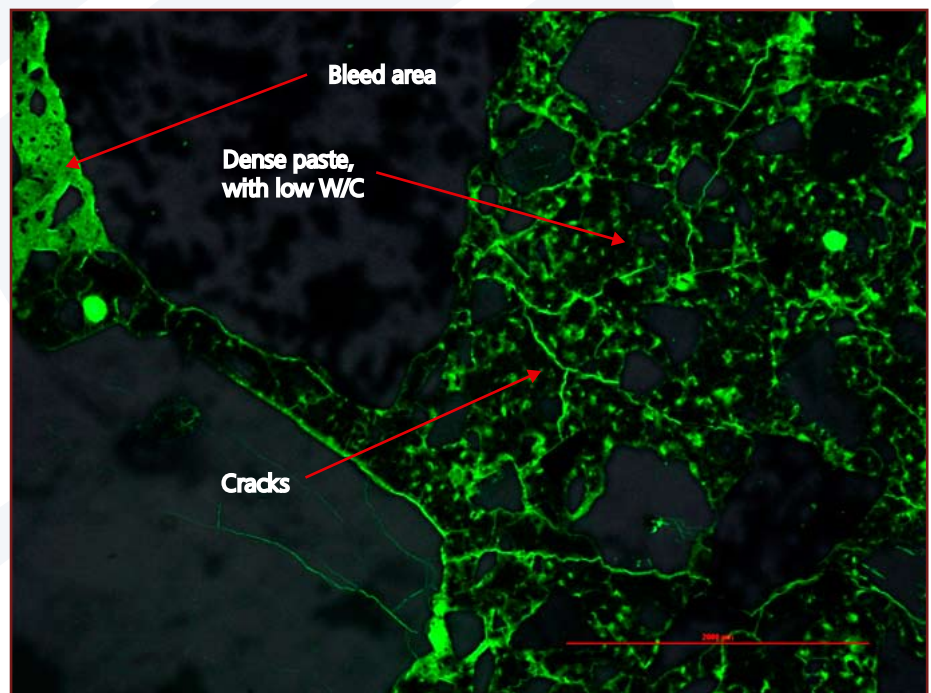
The Concrete Department uses this equipment to analyze the material properties and also to determine the cause and nature of its eventual failures. How these analyses may be helpful for different purposes is described in the articles of this issue of Concrete News. Also the combination of NDT-methods and microstructure analyses are a great advantage to our clients.

The Concrete Department has participated in the exhibition in connection with the 60th anniversary of the Danish Concrete Association. The exhibition, which took place at the Hotel Marriot in Copenhagen at September 20th 2007 is a subject of one of the articles in this issue of Concrete News.

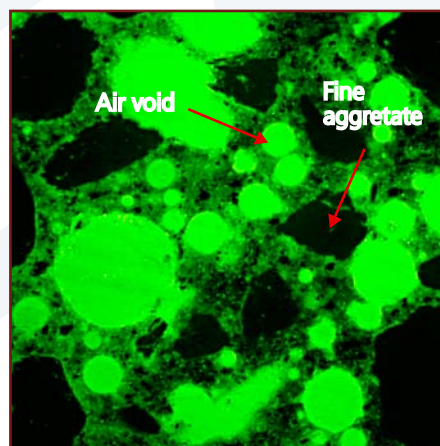
Last but not least we have expanded our department with three new experts, each with their special working fields as described on the last page.

Enjoy the reading.

Brían Kofoed  
Editor



Micrograph is taken from thin section prepared from a blast furnace slag concrete. The image shows numerous micro cracks and areas with bleeding. The image is taken in fluorescent light mode. (fig. 1)



Micrograph obtained in fluorescent light mode. The image shows an extremely high air content in the concrete. The high air content caused a reduction of the compressive strength. The high air content was caused by an overdose of a polycarboylate based plasticiser and an inappropriate air entrainer. (fig. 2)

Getting the right answer to the right time to the right price is always a problem! It is therefore important to have the right tool box available when a problem arises. This goes for the art of medicine as for forensic examination of concrete structures.

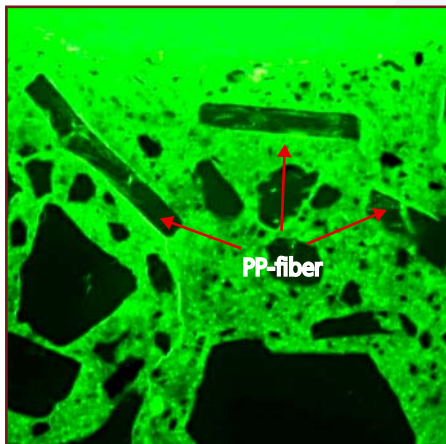
In all kinds of forensic examination it is important to know as much about the patient as possible. In some cases you have the whole story and sometimes you know more or less nothing.

### Methods of examination

Once in a while a tour around the structure with a hammer will solve the problem. In other cases you will scan the whole structure with all kinds of non-destructive techniques going from Geo penetrating radar to X-rays, continuing with seismic methods like impact echo, SASW, MASH and ultra pulse echo.

Then you might take cores or other samples from strategic localities and describe them

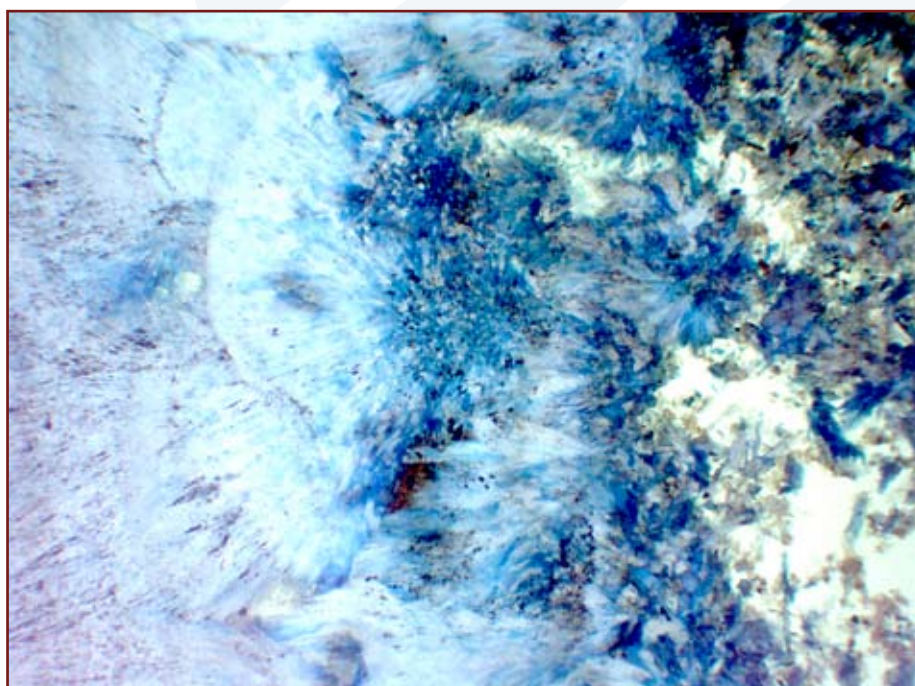
in the laboratory. Occasionally, this will answer the question why the patient died or what is the risk of dying.



Micrograph obtained from the surface of a polypropylene fiber reinforced concrete. Note the fine cracks running in the fiber/paste interface. The sample was taken from a bridge deck which suffer from severe surface scaling. The scaling was caused by freeze thaw. The concrete was inadequately air entrained, the water to cement ration was too high and secondly, the fibers were working as wicks, allowing water and salt to penetrate into the concrete. (fig. 3)

### Sophisticated microscopes

The study of the microstructure of failed materials is often crucial for understanding the failure mechanism. For that reason FORCE Technology is using sophisticated optical and scanning electron microscopy and focused ion beam techniques to help find the cause of failure in metals, composites and concrete.



Optical microscope micrograph obtained in normal light mode. The image shows needle shaped deposits of iron phosphate on a concrete surface from a fermenting tank for domestic sewage sludge. (fig. 5)

### Erroneous recipe?

The reason for failure in concrete structures cannot always be explained by means of NDT or macroscopic analysis. The failure can be caused by inappropriate constituents, erroneous recipe and or deviations from recipe, wrong production methods or external causes like salt, moisture or extreme temperatures.

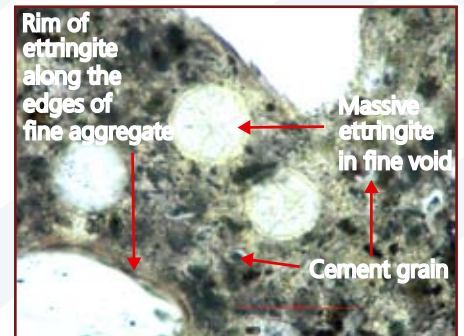
The first choice to solve this problem is by preparing a fluorescent epoxy impregnated thin section of a sample from the failed structure. The thin section is usually 45 mm by 30 mm wide and it is ground to a thickness of 20 micron.

By studying the thin section in normal and polarised light it is possible to describe and quantify the constituents and thereby checking if the concrete was prepared according to the recipe.

By studying the sample in fluorescent light mode it is possible to determine the water to cement ratio, and describe the air void structure and cracking and porosity patterns. (see fig. 1-2)

Optical microscopy will also reveal alkali silica reactions/alkali carbonate reactions, sulphate attack, etching/dissolution, secondary deleterious deposits and carbonation. (see fig. 3-5)

By describing the cracking pattern versus deleterious reactions like carbonation dissolution or precipitation of secondary mineral or salts can help establish when the cracking was initiated.



Micrograph showing fine voids filled with massive ettringite and a rim of ettringite along the edges of the aggregate. This is typical for a phenomena called DEF or Delayed Ettringite Formation. DEF is usually observed in heat cured pre cast elements, where the curing temperature has exceeded 70° C. Ettringite is formed as a natural part of the hydration products, during the normal hydration process, ettringite is not stable above 70° C. When the element cools down and is left in a humid condition ettringite will start to form. The sulphate phases formed above 70° C take up less space than ettringite. When the ettringite starts to form, the paste will expand and thereby weaken the concrete. (fig. 4)

### Scanning electron microscopy

In some cases it is not possible to determine composition of secondary precipitates by optical microscopy alone. In other cases you need to describe the hydration processes and habit of cement or mineral additives like fly ash, slag or other pozzolanic materials.

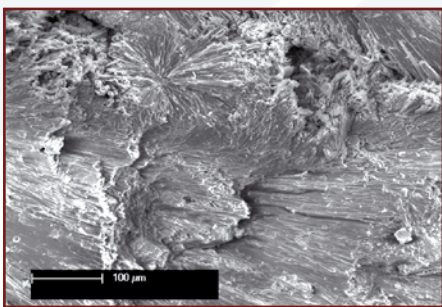
In these cases scanning electron microscopy (SEM) combined with electron dispersive energy detector (EDS) is a strong tool for describing very fine morphological details and local chemical variations. (See fig. 6-8)

Scanning electron microscopy can be conducted on almost any kind of material as long as it will fit into the chamber of the instrument. Before conducting the SEM/EDS analysis it is important to plan what you are looking for, since the area you will be scanning usually is less than 5 mm by 5 mm.

In order to get the best result you will have to choose whether you are going for morphology or composition. You can almost always get both, but you always want to minimize your number of analysis.

### Coated or uncoated samples

When you are searching for fine details on a nanometre scale you will have to coat the sample with a fine layer of gold or platinum. The drawback by metal coating the sample is that it prevents you from getting a proper



SEM image showing solid deposits of iron phosphate on a concrete surface from a fermenting tank for domestic sewage sludge. (fig. 6)

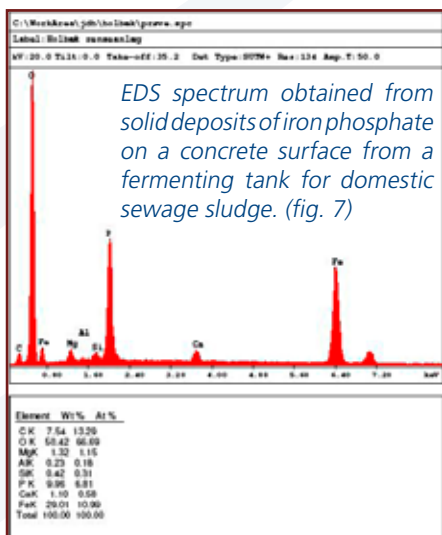
EDS-spectrum from the material below the coating. Most often you will only be able to determine the composition of the coating material.

If you are planning to analyse the sample for a combination of morphology and composition you can in some instances analyse your sample directly without coating it.

Depending on the material, it can be difficult to get detailed images from uncoated samples, since some materials tend to charge when they are hit by the electron beam.

### Electron back scatter

When the aim is to study fine chemical variations in cement grains or secondary mineralisation you will have to perform the



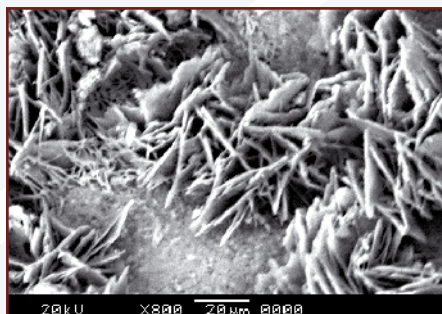
EDS spectrum obtained from solid deposits of iron phosphate on a concrete surface from a fermenting tank for domestic sewage sludge. (fig. 7)

examination on polished samples. Depending on the material, the examination can be performed on uncoated or carbon coated samples.

The survey is conducted in electron back scatter mode where even relatively small variations in the chemical composition will show up as different shades of gray. By means of image analysis it is possible to discern and quantify different phases in a sample.

The different phases in a sample can also be visualised by performing an element mapping, where the backscatter image is combined with EDS measurements.

The system is set up to scan for a series of different elements like Ca, Na, Cl, Mg etc.



Bladed deposits of calcium carbonate on a pigmented concrete paver. The calcium carbonate deposits caused white patches on the red paver. (fig. 8)

The distribution of each element is displayed in a separate window. This method gives a good impression about the distribution of a chosen set of elements in the field of view.

Protective surface coatings and functional nano coatings on almost all kinds of materials are becoming more and more important. Since these types of coatings are very thin (nanometre size) they are also very difficult to describe.

By examining the sample with FIB-SEM it is possible to "dig" into the sample and thereby generating a profile through the surface layer including the nanocoat.

## Conclusion

The study of the microstructure of almost all failed material is important to understand the failure mechanism.

The micro structure alone will not always give the answer to the problem but in combination with the information about the structure and other analytical methods, it can be a strong tool.

For further information:

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## Fact box

FORCE Technology has sophisticated equipment for micro examination material characterisation and testing of almost all types of solid materials. The equipment is used as standard tools in our services and consulting about corrosion, metal characterisation and concrete examinations.

FORCE Technology can assist in customising, planning and execution special examinations of almost any kind of material.

FORCE Technology is accredited for a broad line of material testing procedures according to the EN standards.

## Danish Concrete Association 60<sup>th</sup> anniversary

FORCE Technology was present at the exhibition in connection with the 60th anniversary of the Danish Concrete Association. The exhibition took place at the Hotel Marriot in Copenhagen at September 20th 2007.

### Equipment on parade

We presented our newly released VMM, a voltmeter developed for measuring the potentials of our ERE 20 reference electrodes. Moreover we presented a lot of our equipment to the visitors such as e.g. our GalvaPulse used for determination of



corrosion rate of reinforcement and our recently acquired GSSI SIR-3000 Radar, which can locate details in concrete down to a depth of 0.5 meters and larger objects in the ground down to 4 meters. The equipment was used to investigate the structure of the floor underneath the surface of our booth.

### Demonstration of sensors

Also our sensors for monitoring of reinforcement corrosion, ERE 20 reference electrode and CorroWatch multiprobe were demonstrated by means of a specially constructed concrete mock-up.

# New employees in the Concrete Department

During the past few years the Concrete Department has undergone a very positive development of activities and financial growth.

Due to these circumstances it was decided to extend the actual staff with three new employees, a technician, an engineer and a geologist. Additionally **Peter V. Nygaard** has finished his PhD. study and has now permanently joined the staff of the Concrete Department.

## Our new employees are:

### Dennis Lund Nielsen

35 years old, with a background in construction technique and with experience in construction work and maintenance, the last 7 years responsible for these matters at Herlev hospital near Copenhagen.

Dennis shall assist in the daily work, which includes condition assessment by means of non-destructive techniques and manufacturing/installation of sensors for monitoring of reinforcement corrosion.

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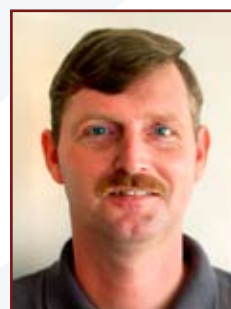


### Peter Vind

40 year old engineer with a background as a construction engineer and with many years experience from the work in contractor companies (mostly abroad on the big construction sites). The last three years Peter Vind worked as a Quality Assurance Manager for company E. Pihl & Søn in a project in Zanzibar on the African East Coast.

The big experience of Peter Vind from construction sites around the world is of great value and will contribute excellently to the competences of the other employees of the Concrete Department. Peter Vind will be situated at FORCE Technology in Aarhus from where he will be able to offer the whole package of products and services of our Concrete Department to our Jutland customers

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### Jens Henriksen

has a background as geologist, M.Sc. from the University of Copenhagen. He has been working with forensic examination of concrete and related materials and natural building material for the last 14 years. His primary fields are micro examination of concrete and other cementitious materials using optical microscopy and SEM/EDS. Additionally he has been working with a range of different NDT techniques for examination of concrete structures.

Prior to his employment at FORCE Technology he worked at RAMBOLL, W.R. Grace in Boston US and Teknologisk Institut in Denmark.

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