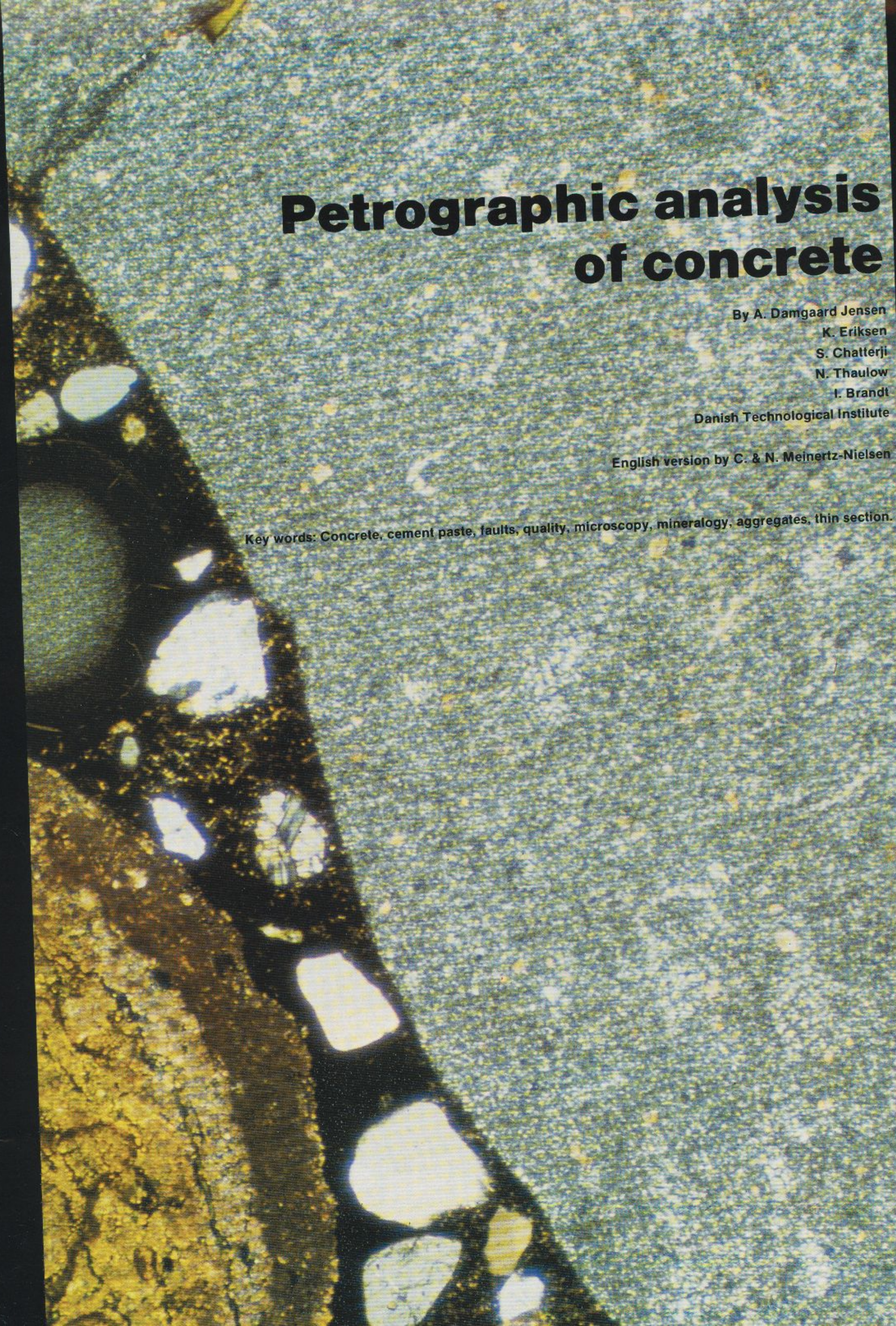

# Petrographic analysis of concrete

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**Hardened concrete bears inherent evidence of the materials used, the methods of work, factors influencing the concrete both during the hardening process and later, chemical transformations and of any damages. Analysis of the concrete's structure and its components, using suitable methods and combination of methods, makes it possible, in principle, to describe the composition of the concrete, account for its origin and determine events which occurred in the concrete during and after its hardening. Application of the same methods makes it possible to evaluate, with reasonable accuracy, the quality of recently cast concrete. On the basis thereof the durability can be predicted through comparison with an idealized model and with the existing empirical information. The latter naturally has to be continually expanded, verified and improved. In visual analysis of structure and composition, usually known as petrographic analysis, the eye is used as a measuring instrument for identification and determination of structure phenomena and material composition e.g. in concrete [1], [2], [3]. The object of this publication is to outline the methods used for petrographic analysis of concrete and the subsequent possibilities for evaluation of concrete quality.**

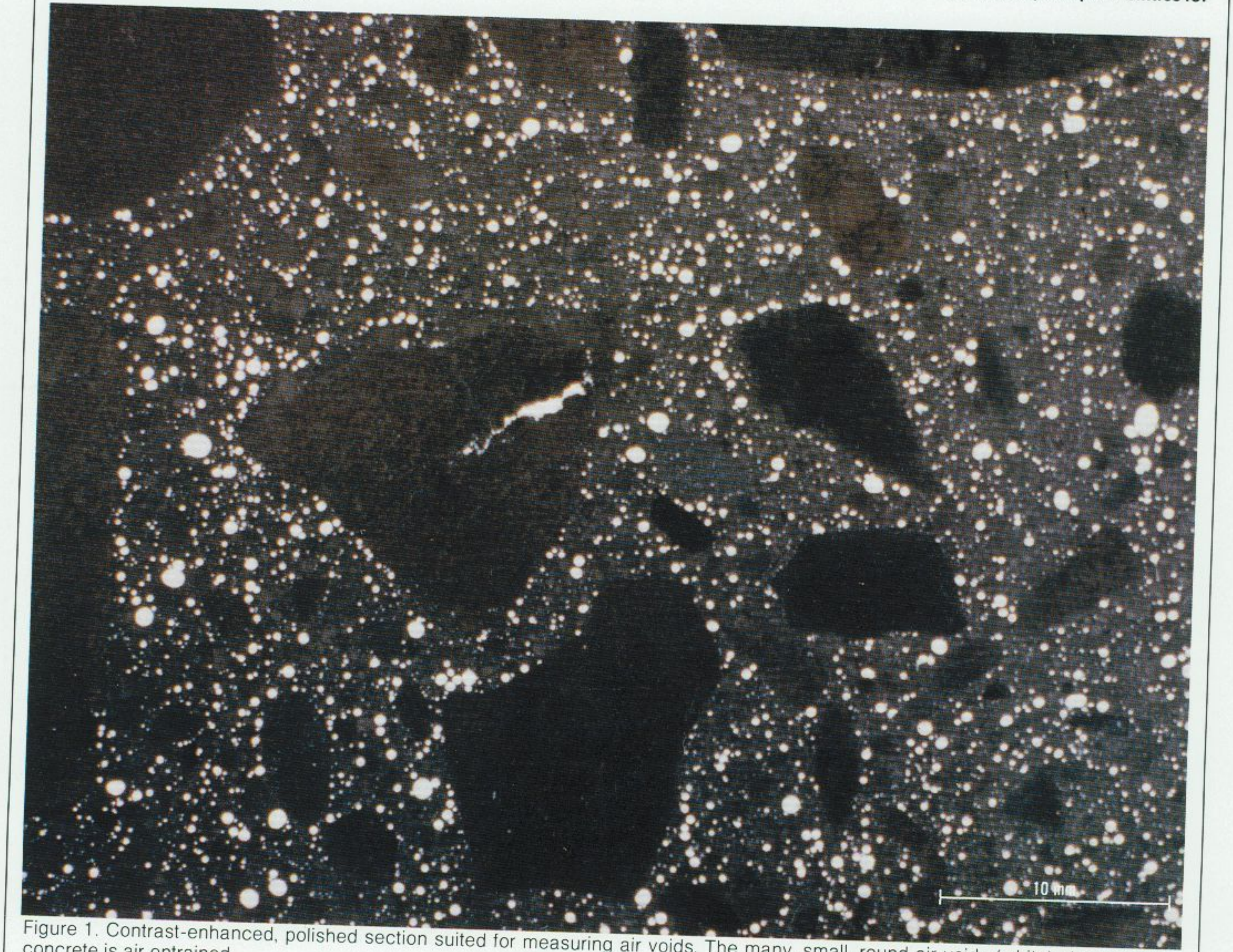


Figure 1. Contrast-enhanced, polished section suited for measuring air voids. The many, small, round air voids (white) show that the concrete is air entrained.

Petrographic analysis of concrete may for instance be used in the following situations:

- Inspection of concrete structures (condition survey).
- Investigation of damaged concrete structures, [4].
- Quality evalution of new concrete products.
- Quality control of new concrete structures, [5].

Similar petrographic analysis can also be used on other materials (e.g. bricks, natural stone).

Depending on circumstances and aims, an analysis of a concrete structure may comprise:

- Visual examination of the concrete structure and selection of locations to be analysed (extraction of concrete cores).
- Visual examination of concrete cores (macro-analysis) and selection of samples for further investigation (micro-analysis, air void determination, crack detection, chloride content etc.).
- Carrying out of these analyses and selecting of samples for supplementary investigation (SEMEX, X-ray diffraction, chemical analysis etc.).

Each individual analysis includes a ***sample preparation,*** i.e. polished section, thin section etc. A number of aids are used (magnifying glass, microscope and occasionally ruler, crack width gauge, phenolphtalein etc.) so that it becomes easy to observe and measure structure and constituents. The sample location and number of samples are chosen according to the purpose of the analysis. It makes a big difference whether the analysis is part of an investigation of deterioration or is part of the quality control of a control section. The number of samples should not be too few since concrete has its random variations in strength, particle distribution etc. Reproducibility and repeatibility of this integrated method have been tested in a number of major construction projects and found to be good.

# Macro-analysis

A macro-analysis is carried out by visual examination of a concrete core or a cut section of the sample. A cut section is more suitable for examination than the coarse surface of a core. The cut surface may be polished. This will enhance the details. The examination is carried out using a magnifying glass, stereo microscope, ruler etc.

The macro-analysis (fig. 2) gives information on:

- Aggregates: particularly mineralogy, quantity, distribution, orientation and shape of the stones. Quantitative determination of the stone content is carried out on the cut section by point count or by the linear traverse technique.
- Paste: particularly its distribution and carbonation depth (using phenolphtalein).
- Air voids: particularly the content of entrapped air indicating the compaction effectiveness.
- Cracks: particularly cracks wider than 0.1 mm and their orientation.

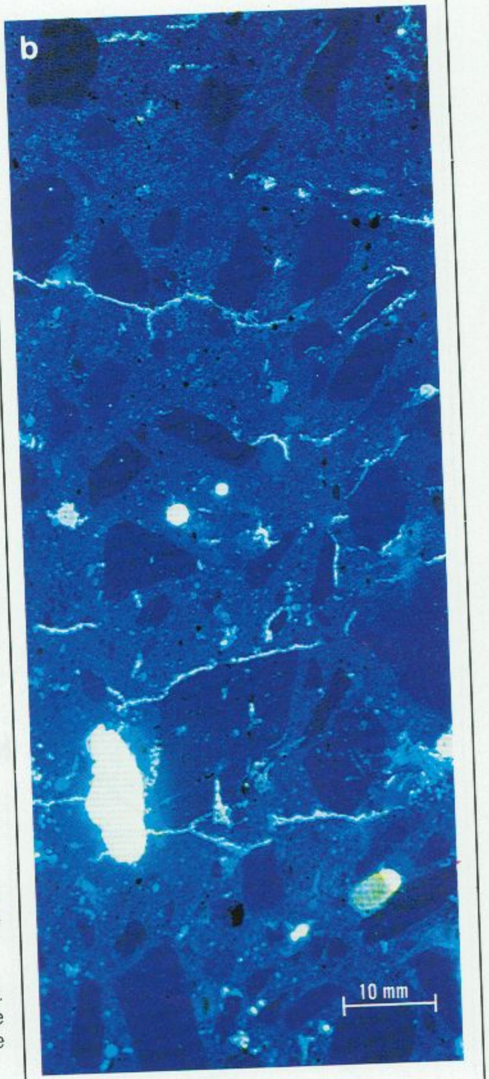
Figure 2
A cut section makes it possible to examine the coarse aggregate, compaction, large cracks etc.

Left: An impregnated, polished section in ordinary light. The original concrete surface is to the left.

Right: Same section in ultra-violet light. Shows several cracks penetrating the sample from the surface as well as one internal crack.

- The surface: texture, deterioration and surface treatment.
- Reinforcement: dimension, position, bond and corrosion, if any.

Specially prepared, polished sections are used for determination of the air void system and for detection of cracks.



**The air void system** is examined on a polished section which is contrast-enhanced making the voids clearly visible for measurement by automatic picture analyser, see fig. 1 [6].
The method determines the air content and the chord lengths of the air voids in the section. Certain assumptions are made and a mathematical calculation gives the specific surface of the air voids (the ratio between the surface area of the air voids and their volume, $mm^2/mm^3$) and the spacing factor (the average distance from any point in the cement paste to the nearest periphery of an air void).
Crack detection is carried out on a polished section impregnated with flourescent epoxy. Illumination of this section with ultraviolet light will clearly show cracks down to 0.02 mm and air voids and differences in

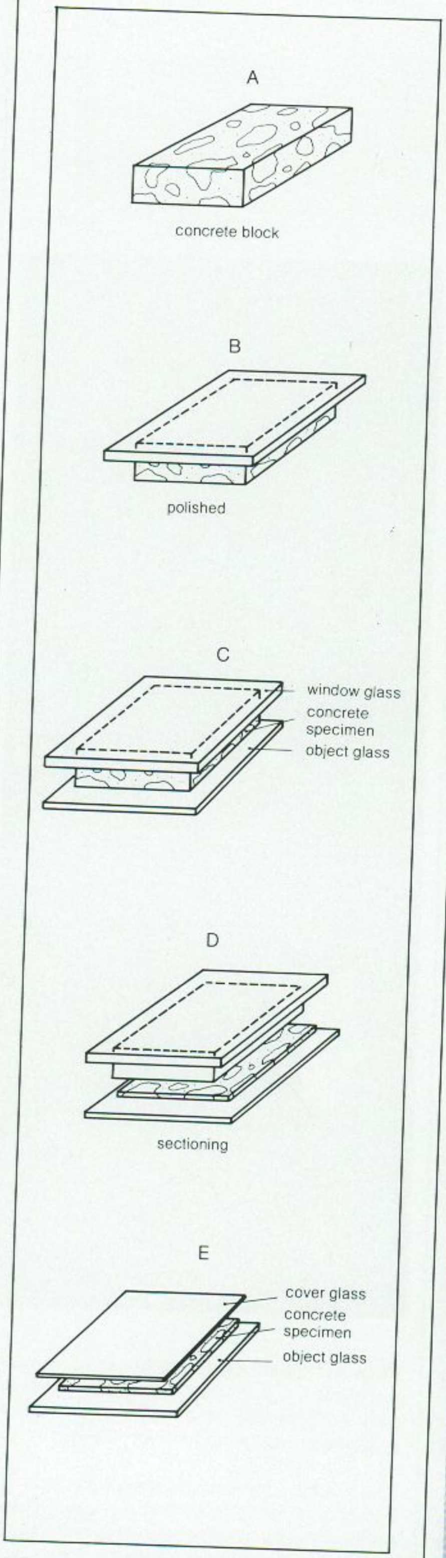


Figure 3. A thin section consists of a thin slice of concrete impregnated with fluorescent epoxy, glued to an object glass and protected by a cover glass. It is processed from a concrete block (A) which is polished on one side (B). After impregnation, the concrete sample is glued to an object glass (C) and cut approximately 1 mm above the glass (D). The concrete slice on the object glass is then ground down to a thickness of 0.020 mm and finally it is protected by a cover glass (E).

A micro-analysis is an examination in a microscope of a thin section made from the concrete sample.

## The thin section

Thin sections have been used for many years by geologists for investigation of rock, minerals etc. Thin sections were made and analyzed in Denmark in connection with a large research project in the 1950s when a working party, for the first time, investigated and described the alkali-silica reactions in Danish concrete [7].

Today, thin sections used for concrete analysis are improved by adding a flourescent dye to the epoxy used for impregnation. As a result porosity and microcracks are made visible.

A thin section is made from a concrete block cut from the relevant position in the sample. The concrete block is usually 50 × 30 × 15 mm. It is soaked in alcohol and dried under vacuum. Drying with heat and in air may produce cracks in the concrete. One of the surfaces of the dry concrete block is polished. The block is then vacuum-impregnated with epoxy containing a yellow fluorescent dye. After hardening of the epoxy the polished surface is freed from excess epoxy and glued to an object glass. The block is then cut approximately 1 mm above the object glass and the concrete slice bonded to the object glass is ground down to a thickness of 20 microns (0.02 mm). At this thickness the concrete is translucent and semi-transparent. Finally, a thin cover glass is glued to the thin section, see fig. 3.

A thin section covers a concrete area of about 50 × 30 mm which means that a thin section is large compared with sand, cement, cement paste, air voids and microcracks in the concrete, but small compared with stones and large cracks.

## The microscope

A laboratory microscope (fig. 4) is used for the examination of a thin section. It may be used both as a polarization microscope and as a fluorescense microscope.

In a polarisation microscope (fig. 5) light is sent through a polarizer where it is plane-polarized, i.e. oscillates in one plane only. The polarized light is then sent successively through the thin section, a second polarizer (usually called the analyzer) to the eyepiece. Here the concrete components (stone, sand, paste, cement grains, air voids, decomposition products etc.) can be examined. If the examination is carried out keeping the second polarizer parallel to the first, the examination is called parallel polarizers (=) mode. If the second polarizer is at right angles to the first, the examination is called crossed polarizers mode (X). In the latter, polarized light from the first polarizer can pass through the second polarizer if its polarization plane has been rotated by its passage through the thin section. Birefringent crystals in the thin section (aggregates, cement grains, calcium hydroxide etc.) will rotate the plane of polarization and will be visible through the eyepiece where they can be identified.

Subsequently a lambda ( $\lambda$ ) plate (gypsum) can be inserted in the light beam. This will cause a change of colour depending on the crystals and their orientation. The use of crossed polarizers and lambda plate makes the identification of many crystals easier.

In the fluorescent microscopy (F) a strong light source is used together with blue and yellow filters. In the eyepiece it can be observed how much light is produced by fluorescense in various areas of the thin section. A visual impression is given of the porosity of each area, i.e. its compactness, cracks and air voids. Photographs taken in the 4 different microscope modes are shown in fig. 6.

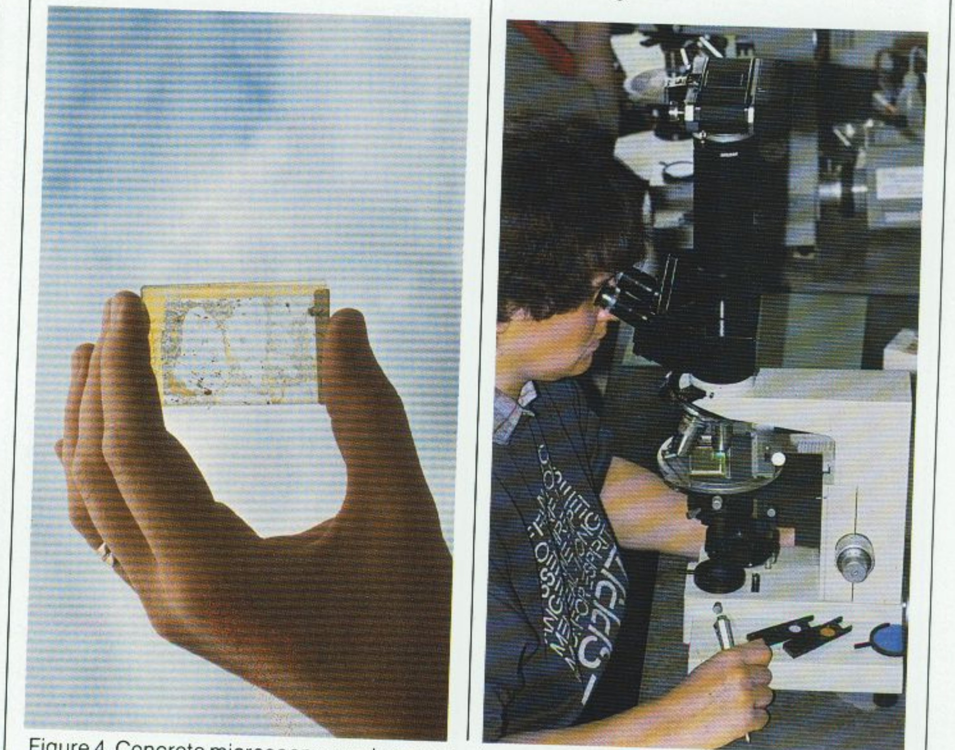


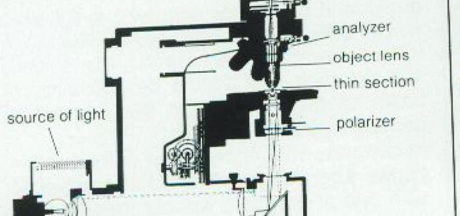
Figure 4. Concrete microscopy needs a good microscope and an experienced petrographer. The left photo shows a completed thin section.

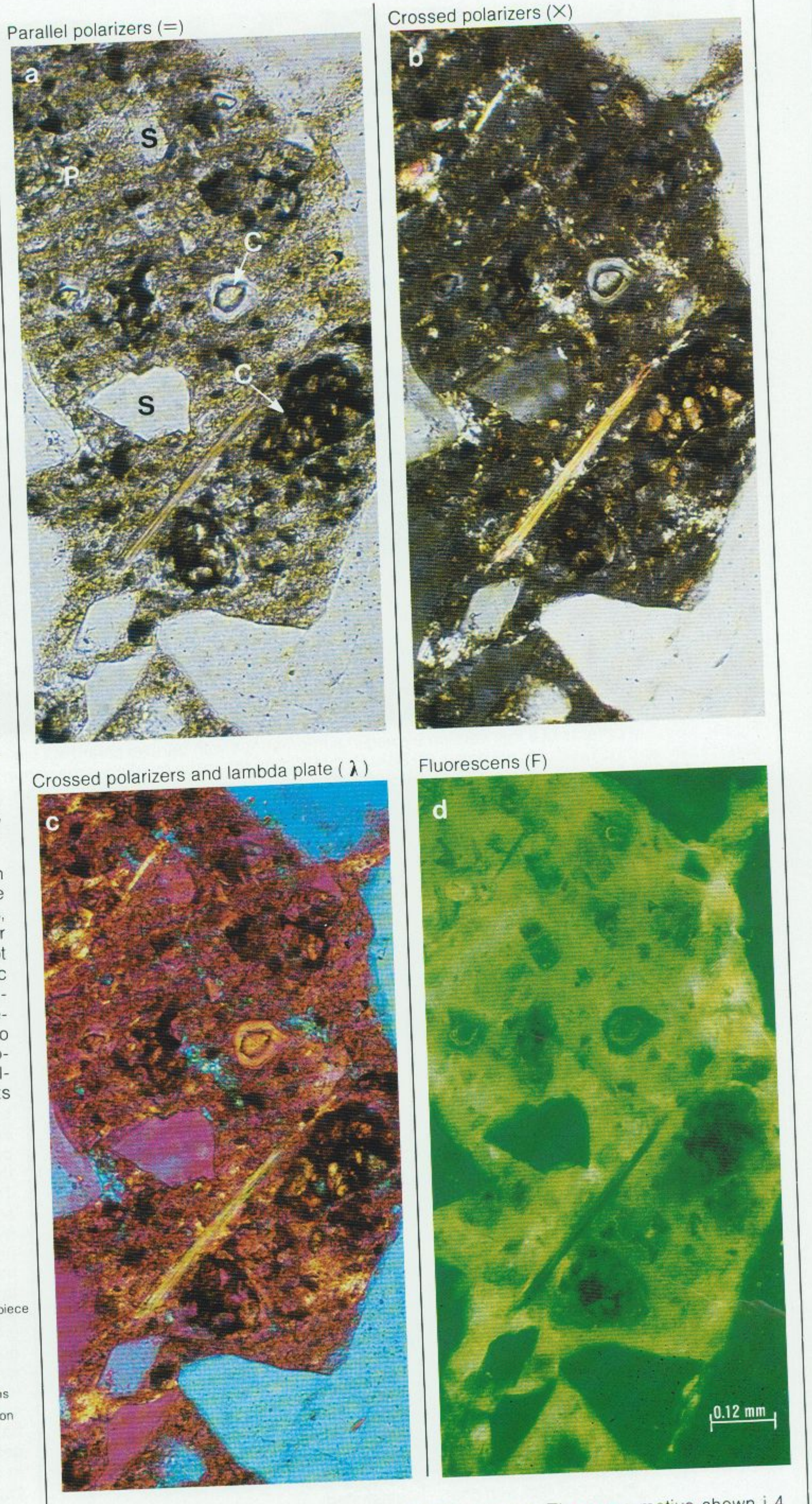
## The micro-analysis

For a general examination of a thin section 25× magnification is used. For a more detailed examination 65× is used. In analysis of cement paste, cement grains, reaction products etc. 160×, 250× and possibly 400× are used, see fig. 7.
The micro-analysis provides information on:

- Aggregates: particularly type and quantity of sand, distribution, filler content, particle shape and extent of alkali-silica reactions.
- The cement-paste, i.e. cement type, degree of hydration, content of fly-ash and micro silica, position of hydroxide (portlandite) formation and carbonation. Furthermore, unusual chemical reactions in the cement paste and bonding of paste with aggregates.
- Paste density , i.e. an expression of the water/cement ratio and the paste homogeneity which will depend on mixing efficiency and dispersion of micro silica, if any.
- Air voids, i.e. types (spherical voids, angular voids, air enclosures), distribution and filling of voids.
- Cracks: particularly fine cracks with a width below 0.1 mm, crack orientation, crack filling and often the cause of cracking (alkali-silica reactions, plastic shrinkage, water separation etc.).
- The surface, i.e. the texture, deterioration, cracks, porosity and surface treatment.

By this technique any phenomena in an area of a size less than 5 microns can be studied (cement grain, various crystals, crack width etc.). Presence of an additive or use of sand containing humic acid can not be seen directly, but the effect of plastic emulsion on the texture of calcium hydroxide can be seen. With sufficient experience such observations can be used to evaluate whether the substances mentioned are present in the concrete. In the following a number of the important elements of micro-analysis are described.




Parallel polarizers (=)

Crossed polarizers (×)

Crossed polarizers and lambda plate (λ)

Fluorescens (F)

Figur 6. Magnification 63× with polarization microscope. The same motive shown i 4

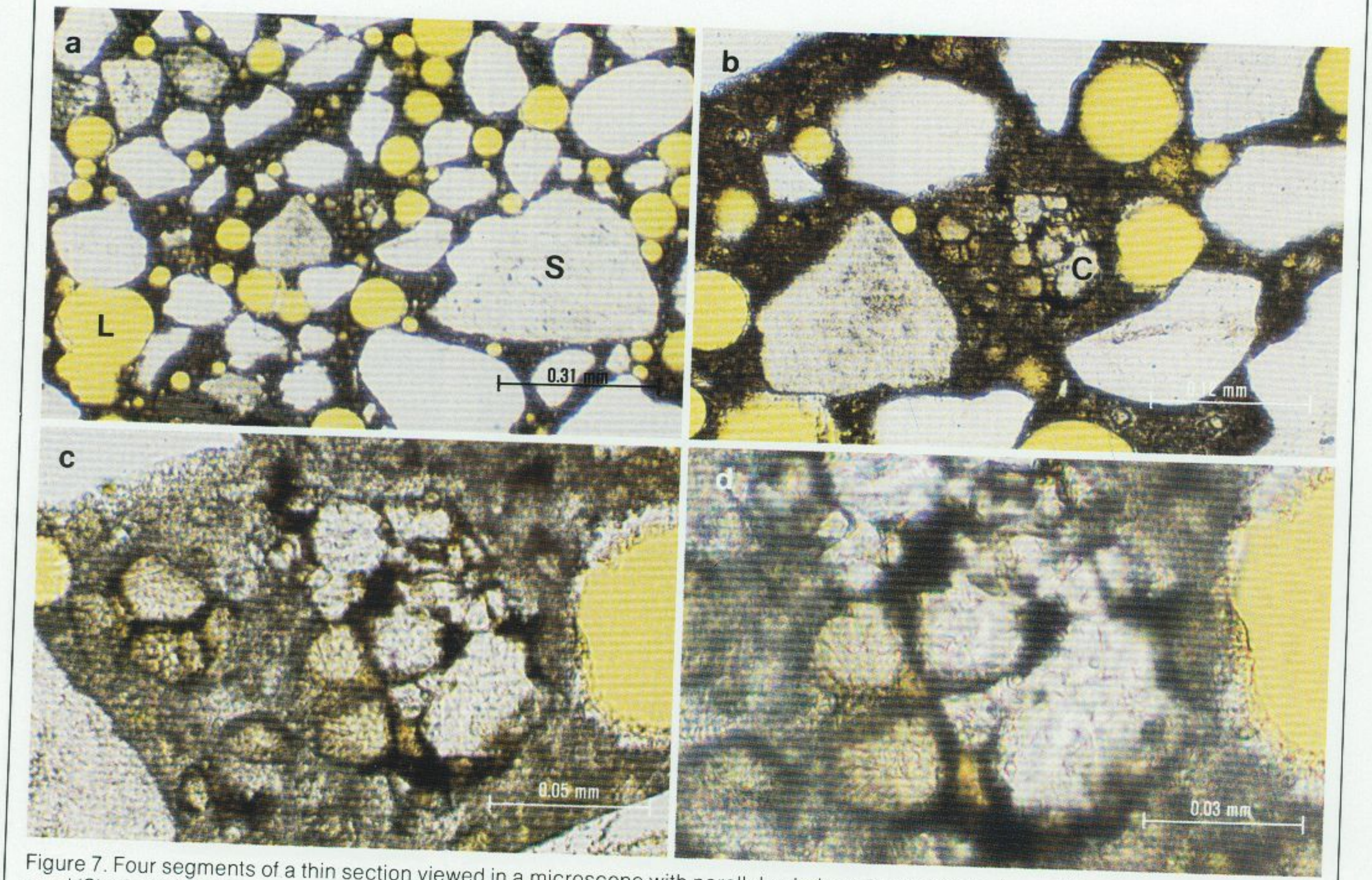


Figure 7. Four segments of a thin section viewed in a microscope with parallel polarizers but at different magnification. (a) 25×. Showing sand (S), air voids (L) and cement paste. (b) 63×. Part of picture (a). In the middle a large cement grain is seen. (c) 160×. The large cement grain and several smaller ones are seen. (d) 250×. The cement grains consist of $C_3S$ (light), $C_2S$ (brown) and $C_4AF$ (black).

## The sand

A normal thin section of concrete contains more than 1000 sand grains, so the analysis is carried out with a reasonably good statistical precision.

Thin section examination of sand in concrete provides information on the mineralogy of the sand. Quartz, felspar and rock fragments are usually predominant. They are usually of little interest. Mica, limestone, shells, clay etc. may be of some interest (see fig. 8). But it is the content and type of flint which is the most important, because flint may cause detrimental alkali-silica reactions in concrete.

Flint of various types is seen, some with chalk and some without. The porosity of the flint is highly indicative of its ability to cause detrimental alkali-silica reactions in the concrete. Flint is usually divided in three classes, each with a subdivision with and without chalk:

- Dense flint with chalcedony
- Porous flint with chalcedony
- Opaline flint (always porous)

The porosity of the flint and its alkali-silica reactivity are seen clearly in the microanalysis. Opaline flint is highly reactive and often causes alkali-silica reaction with ample gel formation. Porous flint with chalcedony may to some extent be alkali-silica reactive. Dense flint is usually non-reactive. Concrete which is damaged by alkali-silica reaction is found with porous flint particles which have reacted and there will be gel-filled cracks and voids. In undamaged concrete, which contains reactive sand, flint particles are often found with reactions and sometimes there is alkali-silica gel in voids and paste but without significant crack formation. The degree of reaction depends on several factors, e.g. the compactness of the concrete, moisture conditions and content of alkali, for instance from de-icing salt (NaCl), see fig. 9.

## The cement paste

The coarse fraction of the cement grains can be examined in a thin section using high magnification. The cement minerals $C_3S$, $C_2S$ and $C_4AF$ can be identified optically. A residue of $C_2S$ (Belite) is nearly always found, even in very old concrete. $C_3S$ (Alite) is sometimes, but not always, transformed into cement gel and calcium hydroxide, see figures 7 and 10. However, the original outline of the grain can still be distinguished. An evaluation of quantities and sizes of the various minerals will usually indicate the type of cement.

**Fly-ash and micro silica.** Presence of fly-ash in the concrete, even in small quantities, can be definitely established (see fig. 11). The quantity can be roughly estimated.

Small quantities of micro silica may be difficult to identify, if it is well dispersed. Larger quantities (about 10% of the cement content) can always be verified, partly from silica agglomerates, partly from changes in the paste structure.

**Paste porosity.** In fluorescent microscopy the cement paste lights up because the capillary pores have been impregnated with fluorescent epoxy and the higher the capillary porosity, hence the w/c ratio, the lighter the cement paste will appear.

The capillary porosity, hence the w/c ratio, of an unknown concrete can be determined by comparison with a set of specimens with known w/c ratio. Table 1 shows that the capillary porosity is very much a function of the w/c ratio, particularly at low w/c ratios. The yellow light is generated by fluorescense and its intensity, as seen with the eye in the microscope, is roughly proportional with the quantity of fluorescent dye, hence the capillary porosity and the w/c ratio. There is significant difference between concretes with $w/c = 0.4$ and $w/c = 0.45$ respectively. The difference is less obvious in concretes with $w/c = 0.6$ and $w/c = 0.65$, see fig. 12.

In new concrete the w/c ratio will be assessed too high and should be adjusted on basis of an estimate of the degree of cement hydration.

In concrete with micro silica or fly-ash the capillary porosity determined is comparable with that of concrete without these additives, but with the same compactness. The result is an »equivalent w/c ratio« which gives basis for calculating the activity factor

Figure 8. 63× crossed polarizers. Showing the different appearance of rock fragments (Bf) and limestone (Kk).

for micro silica or fly-ash in relation to permeability. The activity factor for micro silica is usually about 3 and for fly-ash somewhat below 1.

| w/c | cement paste porosity % |
|---|---|
| 0.40 | 8 |
| 0.45 | 14 |
| 0.50 | 19 |
| 0.55 | 24 |
| 0.60 | 28 |
| 0.65 | 32 |
| 0.70 | 35 |
| 0.75 | 38 |
| 0.80 | 41 |

Table 1. Correlation between the w/c ratio and the cement paste porosity, based on fig. 20 in [8].

**Mortar composition.** The composition by volume of the mortar in the concrete may be determined by point count. Usually 1000 points in the mortar are counted (air, cement paste and sand). The w/c ratio is assessed in 10 areas and an average calculated. Subsequently the quantities of cement and sand are calculated.
With the volumetric composition of the concrete established the compressive strength $f_c$ can be calculated using Féréts formula:

where $k$ = an emperically determined constant (typical value: 280 MN/m$^2$)
$C$ = volume of cement
$W$ = volume of water
$A$ = volume of air

It follows that the strength is determined by the matrix porosity only.
Provided the concrete is uncracked and the air void distribution is reasonable, a good correlation is often found between the calculated $f_c$ and the results found in compression tests on drilled cores [9].

## Air voids

All concrete contains air voids, even when no air entraining agent has been added. These voids are usually assumed to be air-filled. Micro-analysis of concrete shows that this is not always the case.
The voids may be seen filled with the following materials:

Calcium hydroxide

Ettringite

Alkali-silica gel

Calcium carbonate (calcite)

Gypsum

water cured for 28 days after casting, and often in outdoor concrete, see fig. 13.

Ettringite is often found in voids in the concrete. Elements for ettringite formation are leached out of the cement paste and are precipitated in voids. It is a sign of the concrete getting older and weaker. If the concrete is subject to sulphate attack the quantities of ettringite in voids and cracks can get very large, see fig. 14.

Alkali-silica gel in the voids is a consequense of alkali-silica reactions in the concrete. The existance of voids reduces the effect of the expansive reaction, otherwise the gel would be found in cracks.

Calcium carbonate crystals are sometimes found on and near waterlogged concrete surfaces. They are caused by carbonation of calcium hydroxide in water.

Gypsum in the voids is usually an indication of a sulphate attack. Small amounts of gypsum may be found on concrete surfaces subjected to acid rain. The gypsum propably comes from the concrete's inherent content of sulphate (from the cement).
The degree of filling of voids varies considerably and is often limited, but there are examples of a considerable part of the voids in an air entrained concrete being partly filled. Extensive filling of voids will

## Cracks

Cracks may be devided in several groups. It is usual to use the crack width as class reference.

- ***Wide cracks:*** crack width over 0.1 mm
- ***Fine cracks:*** crack width 0.01 - 0.1 mm
- ***Micro cracks:*** crack width below 0.01 mm

Fine cracks and micro cracks are divided into:

- Adhesion cracks which run along aggregate particles (fig. 15).
- Paste cracks which run in the paste
- Cracks in aggregates.

The cracks may be found with a predominant orientation or without orientation.

Cracks may have sharp edges and may pass through sand grains as well as stones. Such cracks are assumed to have formed in the hardened concrete (see fig. 17).

The cracks may also have edges of irregular form and such cracks are assumed to have formed in new concrete, for instance as a result of plastic shrinkage (see fig. 18).

Fine surface cracks are assumed to be formed by drying shrinkage.

Cracks related to alkali-silica reactions (see fig. 9).

Cracks may be fully or partly filled with calcium hydroxide, ettringite, alkali-silica gel, calcium carbonate and gypsum in the same way as air voids. Carbonation following the cracks is often found extending deeply into the concrete (see fig. 19).

Figure 9. 63×. The alkali reactive flint particle in this picture is almost fully dissolved by the reaction and cracks extend into the concrete. Fluorescent micrograph.

Figure 10. 160×. Cement paste with several cement grains (C). Parallel polarizers.

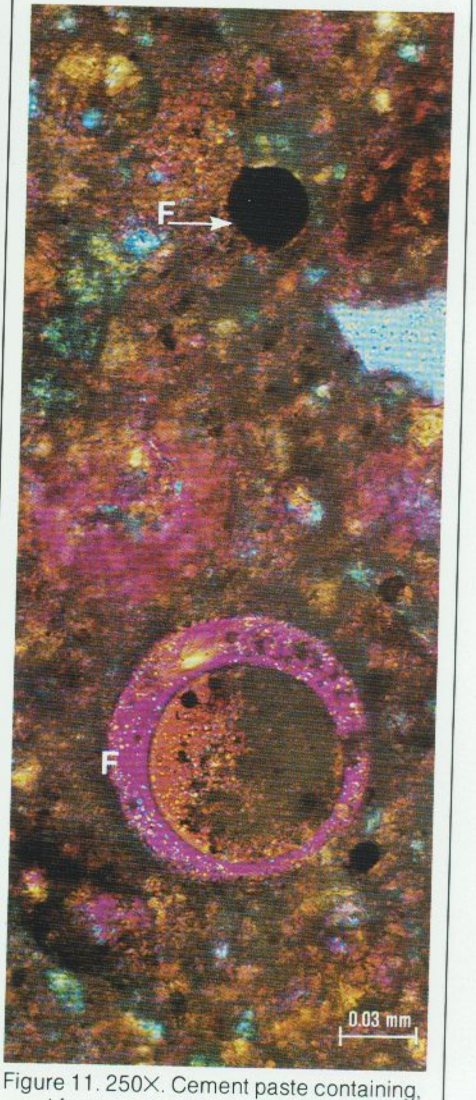


Figure 11. 250×. Cement paste containing, apart from several cement grains, 2 fly-ash particles (F), one being a black coal particle and the other a hollow glass sphere. Crossed polarizers and lambda plate.

# Application

The field of application of petrographic analysis is extensive and is not limited to concrete. It can also be applied to analysis of bricks, repair and building mortars, natural building stone, sculptures etc. and to cores from oil exploration. The main applications of petrographic analysis of concrete are:

- Petrography of sand.
- Investigation of concrete deterioration.
- Evalution of concrete.
- Quality control of concrete.

## Petrography of sand

The content of porous, reactive flint in sand is determined by point count on a thin section made with grains of the sand cast in epoxy containing a fluorescent dye. Usually 2 thin sections are made, one with the 0 - 2 mm fraction and one with the 2 - 4 mm fraction. A maximum permissible content of porous, reactive flint can be specified to secure against detrimental alkali-silica reactions [10].

## Investigation of concrete

Petrographic analysis is used as a routine in investigation of deteriorated concrete structures. The micro-analysis in particular provides a number of useful data on the concrete composition, on the execution of the concrete work and on the effect of deterioration mechanisms. It can be determined whether alkali-silica reaction is a major cause of deterioration, whether sulphate attack (acid environment) has had any influence or whether the concrete has had substantial initial defects (plastic shrinkage cracks, water separation, uneven capillary porosity, early freezing etc.). The difficulties deciding the causes of deterioration usually occur because the concrete can be seen to have cracked, but the reason why is not necessarily apparent. For instance, frost damage in fresh concrete leaves a clear mark whereas frost damage in hardened concrete can not be distinguished from a number of other causes of deterioration.

## Evalution of concrete

Petrographic analysis can be used with advantage to provide relevant knowledge of the inherent structure of the concrete, for instance in connection with development of new types of concrete, e.g. concrete with micro silica and fibre concrete - and in connection with testing and research concerning concrete technology.

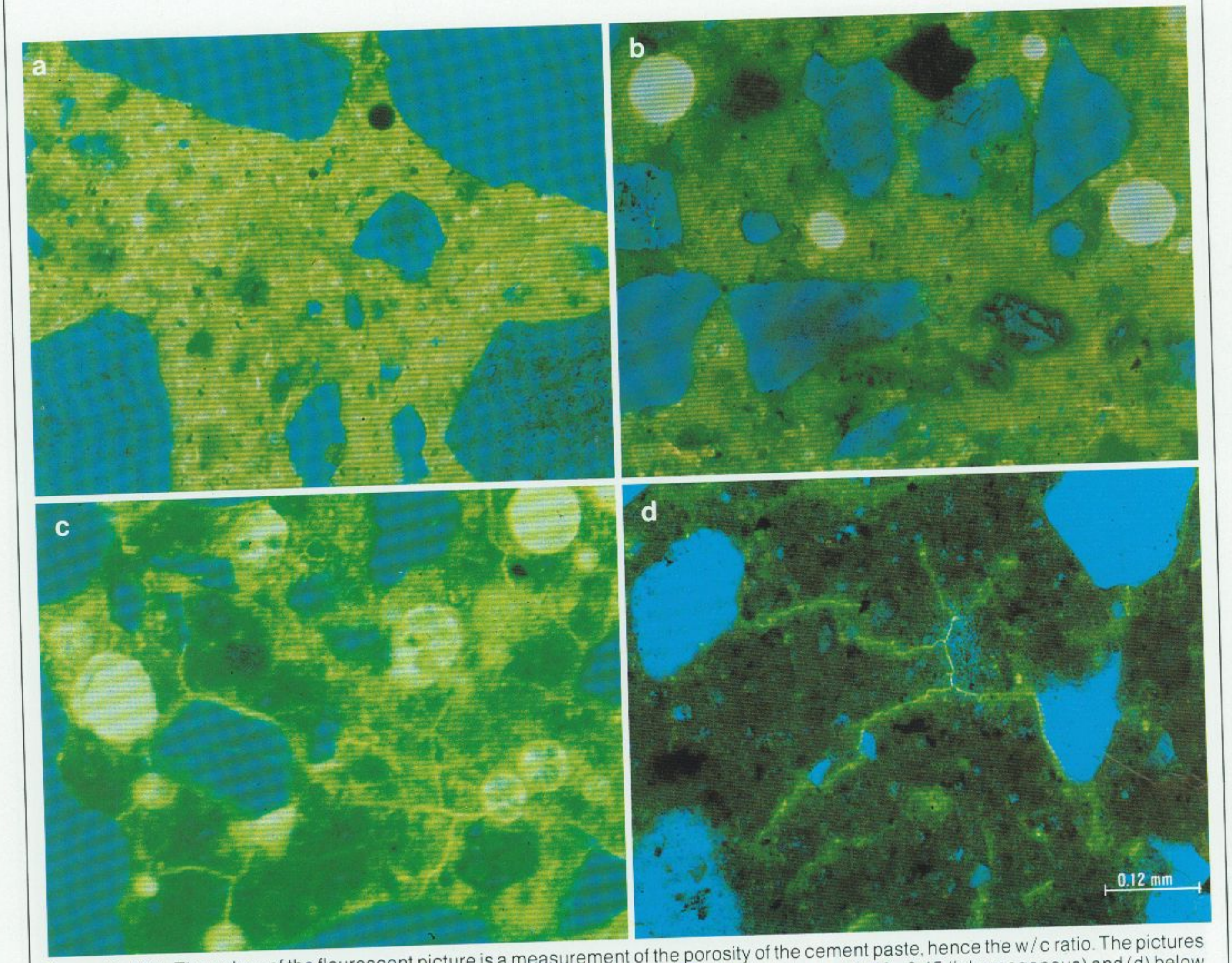


Figure 12.63X. The colour of the flourescent picture is a measurement of the porosity of the cement paste, hence the w/c ratio. The pictures a - d show porosity equivalent to the following w/c ratios: (a) about 0.60, (b) about 0.50, (c) about 0.40 - 0.45 (inhomogenous) and (d) below

## Quality control of concrete

Based on experience it is possible to set up a model for the composition of concrete with good durability:

- Durable concrete consists of a system of good and well-distributed aggregates in a compact and well-hydrated cement paste. The concrete must be homogenous at all levels. In a climate with periods of frost it should normally contain a homogenous system of entrained, small air voids. After being placed in the structure it must be without cracks and signs of abnormal chemical changes. The concrete must be well compacted.

The use of fluorescense impregnated thin sections for evaluation and analysis of concrete, particularly its cracks and porosities, facilitates a registration of a number of details of the structure of concrete including various flaws.
The micro-analysis should preferably be carried out on thin sections which include the original free surface of the concrete. The thin section is examined for major flaws and the homogeneity is evaluated. The material composition of the concrete is checked. The surface is examined with regard to flaws in a number of areas and deviation from the internal structure is described. The evaluation of the internal structure comprises the following:

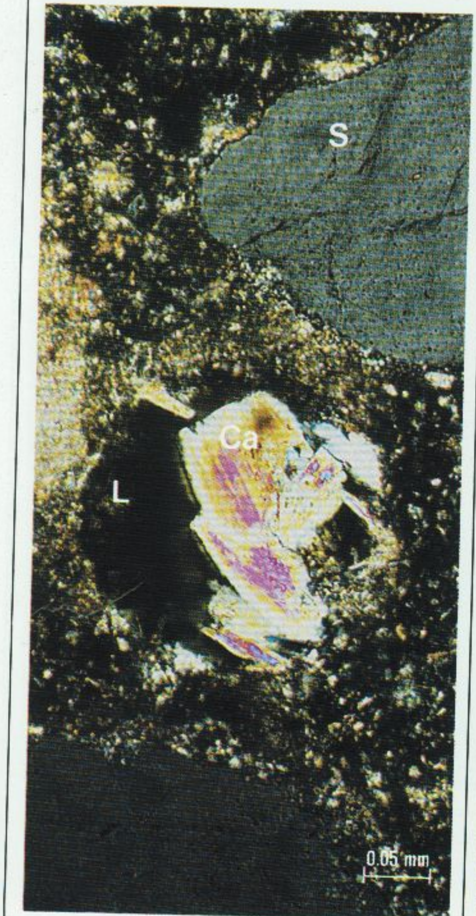
- Cracks in the paste (micro cracks).
- Adhesion cracks (micro cracks).
- Agglomerates of air voids.
- Capillary porosity.
- Uniformity of the capillary porosity.

Fine and wide cracks together with other larger defects are registered during the examination of the thin section as described above.
Depending on the quantities of cracks, flaws etc. the concrete will be approved/-not approved with respect to durability.

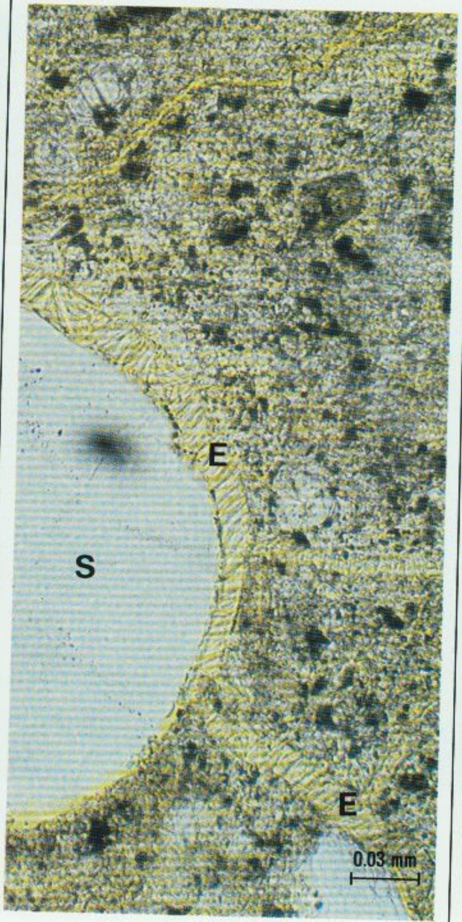
If the concrete contains an excess number of cracks, flaws etc. directions can be given about changes in the production in order to produce concrete with fewer faults. Such changes could be: better mixing, better compaction, better covering or changes in the concrete mix design in order to make the concrete more stable.

Some faults, e.g. unsuitable sand, addition of extra water on site, lack of protection of newly cast concrete, unsuitably prepared construction joints, unsuitable type of cement, excess water etc. are easily detected in the micro analysis.

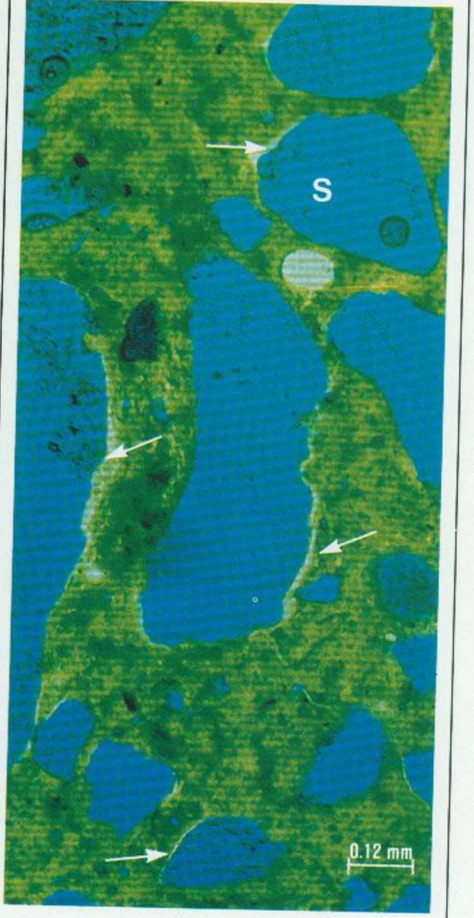
Figur 13. 160×.Void (L) partly filled with coarse calcium hydroxide crystals (Ca). Small calcium hydroxide crystals are seen in the surrounding paste. Crossed polarizers.



Figur 14. 250×. Paste cracks and adhesion cracks (micro cracks) are filled with ettringite (E). Parallel polarizers.



Figur 15. 63×. Showing adhesion cracks along stone and sand particles (arrows). The cement paste is rather inhomogenous (varying colour). Flourescent micrograph.



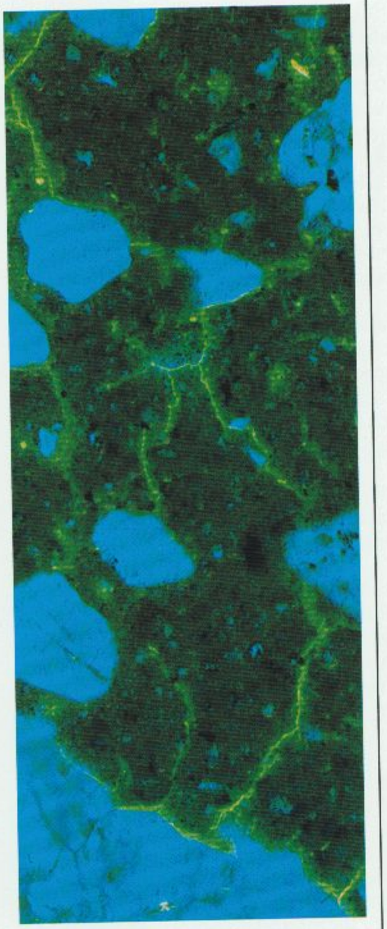


Figure 16. 63×. Micro cracks (mostly in the paste) in a dense (dark) cement paste. Fluorescent micrograph.

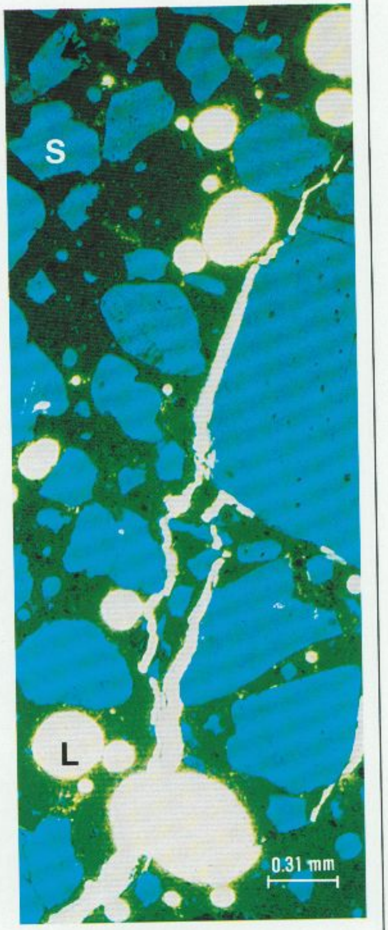


Figure 17. 25×. Fine cracks in a dense (dark) cement paste. The cracks are sharp and jagged. They were formed in a hardened concrete. Fluorescent micrograph.

Figure 18. 25×. Wide, very irregular cracks, presumably

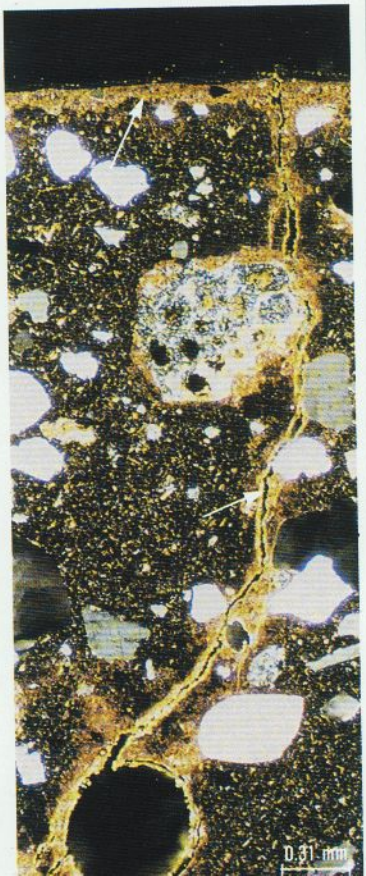


Figure 19. 25×. Concrete with less than 0.1 mm surface carbonation (arrow). The carbonation is also following a fine

| | |
|---|---|
| Agglomerate: | 3 or more air voids in close contact. |
| Alkali-silica gel: | A gel which usually is clear, transparent and non-crystalline, although in some cases it may crystallize. Formed by reaction between porous silica (reactive sand), alkali (from cement, de-icing salt, sea water) and calcium hydroxide. Expands during water absorption. |
| Bleeding: | Water separation in the concrete. Seen as narrow voids along the border line between aggregate and cement paste. Calcium hydroxide may be deposited in the voids. |
| Calcium carbonate: | $CaCO_3$, calcite, found as deposits in voids and cracks and on the surface. |
| Calcium hydroxide: | $Ca(OH)_2$, portlandite. Hydration product of $C_3S$ and $C_2S$. |
| Carbonation: | Transformation of the calcium containing constituents of the concrete by reaction with the carbon dioxide in air. Calcium hydroxide is transformed to calcium carbonate and consequently the paste becomes chemically neutral. |
| Cement: | The visible cement minerals are: $C_3S$, $C_2S$ and $C_4AF$. $C_3A$ is not visible. |
| Cement paste: | Compound of hydration products from the cement-water reaction and unhydrated cement. Micro silica and fly-ash is usually treated as part of the paste. |
| Entrained air: | Usually defined as the small and medium sized, spherical air voids with maximum size 0.5 - 1 mm. |
| Entrapped air: | Comprises irregular and angular air voids of all sizes. |
| Ettringite: | Needle sharp crystals of calcium sulpho aluminate hydrate produced by constituents in the cement paste, including gypsum. It is also produced by sulphate attack on the concrete. |
| Filled voids: | Air voids with complete or partial filling with e.g. alkali-silica gel, ettringite, calcium hydroxide, calcium carbonate or gypsum. |
| Gypsum: | $CaSO_4 \cdot 2H_2O$, calcium sulphate which is found as a constituent of cement. It is also produced by acid attack (sulphuric acid) on concrete. |
| Hydration: | Reaction between cement and water, producing cement gel (cement paste, cement glue) serving as an adhesive in the concrete. |
| Plastic shrinkage: | Shrinkage caused by strong drying of newly cast concrete. The result is often plastic shrinkage cracks in the concrete surface. |
| Plastic settlement | Settlement of fresh concrete, often causing plastic settlement cracks. |
| Re-crystallisation: | Dissolution of crystals and re-depositing either as the same mineral in a different form or as new minerals, mostly under influence of water. |
| w/c ratio: | The ratio of water to cement by weight in the cement paste. If the cement paste contains fly-ash or micro silica these materials can be taken into account with the cement using an activity factor and an equivalent w/c ratio can be calculated. |

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