CONCRETE, HARDENED:
AIR VOID STRUCTURE AND AIR CONTENT

Key words: Concrete, test method, air content

1 SCOPE AND FIELD OF APPLICATION
The method can be applied to determine the air content and air void structure in hardened concrete. The air void structure is described by means of the parameters, air void content, specific surface and spacing factor. Two methods are described in the standard, the linear traverse method and the modified point count method. The measurements can be performed on either face ground specimens or thin sections.
The method can be applied to test the air entraining agents, and to evaluate the quality of the air void structure in the hardened concrete.

2 REFERENCES
NT BUILD 191 Concrete, fresh: sampling.
NT BUILD 202 Concrete, hardened: sampling and treatment of cores for strength tests

3 DEFINITIONS
Air void, a small space enclosed by the cement paste in concrete and occupied by air.
Entrained air, air voids with a diameter 0.008-2 mm.
Entrained air, air voids with a diameter greater than 2 mm.
Air void content, \( A \), the proportional volume of air voids in concrete expressed as volume percentage of the hardened concrete.
Specific surface, \( \alpha \), the surface area of the air voids, related to their volumes, in hardened concrete, expressed in \( \text{mm}^{-1} \).
Spacing factor, \( L \), an index related to the maximum distance of any point in the cement paste from the periphery of an air void, expressed in \( \text{mm} \).
Paste content, \( P \), the proportional volume of cement paste in concrete expressed as volume percentage of the hardened concrete.
Chord, the intercept length across an air void or an aggregate, expressed in \( \text{mm} \).

4 SAMPLING
If no sampling procedure is described in the test report the sampling is as stated in NT BUILD 191 or 202.

5 METHOD OF TEST
The measurements can be carried out on face ground specimens or on thin sections according to two methods, linear traverse method or modified point count method. The measurements can be performed as manual, semi automatic or automatic analysis. The spacing factor can be calculated by use of the measured paste content or by use of the paste content calculated from the mix proportions.

Linear traverse method
Measurement is carried out by registering the number and length of chords greater than 0.008 mm within air voids and aggregates along a test line. The total length of the chords related to the total test line determines the volume of air voids in the test sample. By use of these parameters it is possible to calculate the air void content, specific surface and the spacing factor.

Modified point count method
Measurement is carried out by measuring the total number of sections of air voids intersected along a line of traverse and the frequency with which regularly spaced points, (index point), on the line of traverse are superimposed on the sections of air voids and aggregate. By use of these parameters it is possible to calculate the air content, specific surface and the spacing factor.

5.2 Apparatus
5.2.1 Equipment for preparation
The listed equipments are examples of equipment for sample preparation. Other equipments may be used if suitable specimens can be obtained.
- Equipment for marking, cleaning and drying.
- Diamond saw for cutting specimens.

Equipment for preparation of face ground specimens
Concrete saw
Grinding machine
Silicon carbide powder, grain size approximately 120, 60, 30, 16 and 12 µm
Glyserol
Nail hardener
Acetone
Stamp ink, black dull, not water soluble
Zinc paste
Gypsum powder, grain size < 3 µm
Steel scarper
Refrigerator
Oven

Equipment for preparation of thin sections
Diamond saw
Drying chamber
Vacuum chamber
Face grinding machine with diamond impregnated disc for coarse surface grinding
Face grinding disc with diamonds grain size 25-30 µm, for final surface grinding
Epoxy resin and epoxy hardener with fluorescent dye
Ethanol
Microscope slide minimum 28 x 48 mm²
Cover glass
Canada balsam or equivalent

5.2.2 Apparatus for measurements
Microscope or equivalent with at least 50x magnification.
Linear traverse device with an accuracy in length determination of 3 µm.
Point count device or grid for modified point counting.
Equipment for automatic analysis with an accuracy in length determination of 3 µm.

5.3 Preparation of test samples
The test samples may be cast samples or drilled cores from site.

The analysis is carried out on a slice cut out perpendicular to the upper surface of the concrete. The test area must be at least 3000 mm². If the spacing factor is calculated by use of the paste content calculated from the mix proportions, the test area shall be at least 7000 mm². It may be necessary to use more than one specimen (sub-samples) for one measurement to obtain the required test area. The results from different sub-samples are considered to be measurements from one sample.

The air void structure caused by an air entraining agent can be determined by measurement of the air void structure of a cast sample. In such a case the test area should be placed on three slices, located at the top, centre and the bottom of the cube/cylinder. The distance between the edge of the specimen and the slice shall be approximately 6 mm. In all other cases the test area should be placed as near as possible to the upper surface of the concrete. The distance between the slices shall be at least 50 mm.

5.3.1 Preparation of face ground specimens
The sample is ground for about 5 minutes on the grinding machine under continuous addition of abrasives mixed in glycerol and water. The sample and the grinding wheel are then cleaned by using a brush and washed under running water. After washing the sample is rotated 90° counter-clockwise. Grinding is carried out using each powder grade to produce a plane surface of good quality.

The grinding powder of grain size 16 microns may be omitted when the cement paste is strong. Before each change of powder grade, the surface should be checked for defects and wear of the cement paste. The surface may be considered satisfactory when the edges of the air voids are sharp and well defined.

The surface must be without scratches. Air voids are seen as round shaped cavities. Missing grains (torn out during grinding) show cavities with sharp, irregular edges. If the cement paste is weak, it may be strengthened in two different ways:

1. Storage in hot water up to 80°C or
2. Nail hardener is applied to the surface before the final stage of grinding. The nail hardener must be removed completely with acetone before measurement.

The sample can be treated with a special preparation to produce a better contrast between the air voids and the cement paste. This is done by applying ink to the surface from a stamp pad or roller. Care must be taken to prevent the ink from sinking into the air voids.

The sample is then placed in the oven at 50°C for 4 hours. It is then covered with zinc paste and the excess zinc paste is removed after refrigerating. Finally, the surface is covered with gypsum powder which is pressed into the zinc paste filled air voids. The excess gypsum powder is removed with a scraper.

5.3.2 Preparation of thin sections
The following procedure is an example of the method of preparation. Other methods can be used as long as a thin section with the same thickness and quality is obtained.
Cutting of the specimen

A specimen minimum 40 x 20 x 10 mm³, see figure 1, is cut with a diamond saw and glued with epoxy on a microscope slide (working glass). The specimen is then face ground, see figure 1.

Drying the specimen

The specimen is dried in alcohol or in a drying chamber overnight at 30-40 °C.

Impregnation

The specimen is evacuated in a vacuum chamber at maximum 0.05 Bar for approximately 10 min. The epoxy, prepared in advance, is added and the vacuum is maintained for another 10 minutes. The specimen is then stored in a plastic bag in a normal atmosphere until the hardening of the epoxy is complete.

Grinding of the lower surface of the thin section

The specimen is ground until the edges of the air voids are sharp. The paste should, however, still be fully impregnated with epoxy. To ensure that the surface is satisfactory the grinding should be carried out in two steps. First there should be face grinding to achieve an absolute plane surface, followed by final grinding on the grinding disc to achieve a smooth surface.

Mounting on microscope slides

The specimen is glued on a microscope object glass slide with the ground face towards the slide, see figure 2.

Grinding of the upper surface of the thin section

The specimen is cut approximately 1 mm above the glass slide, see figure 2.

The upper surface of the thin section is face ground on the face grinding machine until the thickness of the specimen is about 0.05 mm. Gridding is then continued on the grinding disc until the thickness is 0.025 mm. The thickness is checked by use of bi-refringent colours of common minerals such as quartz and feldspar. It is very important that the thickness is the same in all parts of the thin section.

Finishing the thin section

The cover glass is mounted on the specimen with Canada balsam, see figure 3.

5.4 Measurements

The measurement can be performed as manual, semi automatic or automatic measurement according to three methods.

Linear traverse method

The sample is mounted on the stage of the microscope. The measurement is carried out, at minimum 50x magnification, along parallel lines evenly distributed on the prepared surface. During the measurement, the following parameters are registered:
- Total length of the line of traverse (\(l_t\))
- Total length of chords within air voids (\(l\))
- Total length of chords, within air voids, with chord length <2 mm (\(l_a\))
- Total number of air voids intersected (\(N_I\))
- Total number of air voids with chord <2 mm intersected (\(N_a\))
- Total length of chords in cement paste (if the cement paste content is measured) (\(l_p\))

The line of each traverse should be at least 2 mm. The beginning of each line shall not be measured. The total length of traverse should be at least 2500 mm.

Modified point count method

The sample is mounted on the stage of the microscope. During the measurement, the following parameters are registered:

- Distance between each index point (\(l\))
- Total number of points (\(S_t\))
- Total number of lines of traverse (\(N_T\))
- Total number of air voids superimposed by the index points (\(N_{ai}\))
- Total number of air voids superimposed by the index points where the chord of the air void is < 2 mm (\(N_a\))
- Total number of air voids with chord < 2 mm intersected (\(N_{ai}\))
- Total number of points in cement paste (if the cement paste content is measured) (\(N_p\))

The distance between each index point should be 0.5-5 mm and the minimum number of points should be 1500 evenly distributed over the surface. The starting point shall not be counted. The total length of the traverse should be at least 2500 mm. The total area of measurement should be at least 3000 mm². If the paste content is not measured in the same operation the test area shall be at least 7000 mm².

Automatic image analysis

The automatic image analysis is carried out as modified point counting. The distance between the picture elements shall be maximum 4 \(\mu m\). The number of points shall be at least 250 000. The total area of measurement should be at least 250 mm² evenly distributed over a test area of 3000 mm². If the paste content is not measured in the same operation the test area shall be at least 7000 mm².

5.5 Calculations

The air void structure is described by the air void content, the specific surface, and the spacing factor. In the following the calculations for linear traverse analysis and modified point counting are described. These calculations are also valid for automatic image analysis.

The air void content, \(A\), specific surface, \(\alpha\), and cement paste content, \(P\), are calculated according to the following formulas.

**Linear traverse method:**

\[
A = 100 \frac{l_a}{l_t} \quad \% \\
\alpha = \frac{4N_a}{l_a} \quad \text{mm}^{-1} \\
P = 100 \frac{l_p}{l_t} \quad \% 
\]

**Modified point count method:**

\[
A = 100 \frac{N_a}{S_t} \quad \% \\
\alpha = \frac{400 N_{ai}}{(S_t - N_a) \times A} \quad \text{mm}^{-1} \\
P = 100 \frac{N_p}{N_t} \quad \%
\]

The spacing factor, \(\bar{L}\), is calculated according to the following formulas.

\[
\bar{L} = \begin{cases} 
\frac{P}{A} \alpha \\
3 \left( \frac{1.4 \left( \frac{P}{A} + 1 \right)^{1/3}}{\alpha} \right) \end{cases} \quad \text{mm} 
\]

Where \(P\) is the percentage by volume of air free cement paste measured or calculated from the mix proportions.

The content of entrapped air is calculated according to the following formulas.

**Linear traverse method:**

\[
A_t = 100 \frac{(l - l_a)}{l_t} \quad \% 
\]

**Modified point count method:**

\[
A_t = 100 \frac{(N_{ai} - N_a)}{S_t} \quad \%
\]

5.6 Expression of results

The air content is stated as the percentage by volume of the concrete calculated to the nearest 0.1 %. The specific surface is stated as the surface of the air voids related to their volumes calculated to the nearest mm⁻¹. The spacing factor is stated as the maximum distance of any point in the cement paste from the periphery of an air void calculated to the nearest 0.01 mm.
The cement paste content is stated as the percentage by volume of cement paste calculated to the nearest 1%.

The content of the entrapped air is stated as the percentage by volume of the concrete calculated to the nearest 0.1%.

6 REPORT
The test report shall include the following information, if relevant:

a) Name and address of the testing laboratory
b) Identification number of the test report
c) Name and address of the organization or the person who ordered the test
d) The purpose of the test
e) Method of sampling and other circumstances (date and person responsible for the sampling)
f) Name and address of manufacturer or supplier of the tested object
g) Name or other identification of the object
h) Description of the tested object
i) Date of supply of the tested object
j) Date of the test
k) Test method
l) Any deviation from the test method
m) Test results
n) Inaccuracy or uncertainty of the test result
o) Date and signature