AMAS Workshop on Image Analysis in Investigation of Concrete
SIAIC'02 – (pp.15–25) – Warsaw, October 21-23, 2002.

Application of stereological analysis to quantitative assessment of geometric structure of air-entrained concretes

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1. Introduction

Concrete can be viewed as a two component material, composed of aggregate grains embedded in cement paste. In air-entrained concrete a third component must be distinguished: the air voids introduced into the paste, i.e. pores. Both the aggregate grains and the pores can be treated with regard to their geometry as convex bodies, whereas the paste represents a multi-connected component packing up the remaining region of a specimen of concrete sample. In the analysis which follows the other components of concrete will be neglected.

The pores have two specific properties. The first concerns their shape: they are almost perfect spheres. The other refers to their arrangement in the paste: they occur inside the paste, sporadically adhere to the surface of contact between the paste and the aggregate grains. Under spatial arrangement of grains forming a component we shall understand their location and orientation,

The geometrical structure of air-entrained concrete is made up of a set of pores and a set of aggregate grains embedded in paste as a non-grained matrix. The substructure formed by the set of pores is of special importance, since the pores, their size distribution, number and arrangement in the paste have substantial influence on the freeze-thaw resistance of concrete. A quantitative assessment of the concrete structure is made by means of the method of section of stereological analysis. Let us recall its most important elements. We shall use the notation commonly used in stereology, different from those used traditionally in the stereology of concrete.

Let us consider a specimen of a two component material filling a special region R of the volume V_R. The region R contains a set of convex grains $\{G_i\}$, $i = 1, \ldots, N$, belonging to one component embedded in the other component – the matrix. The geometry of a single grain G is characterized by means of its four basic functionals (Hadwiger, 1955). These are:

- volume V,
- surface area S,
- total mean curvature M,
- total Gaussian curvature C.

Accordingly, to a single grain correspond four (non-negative) numbers:

$$G[V, S, M, C] \tag{1.1}$$

with their dimensions forming a decreasing sequence

$$[l^3, l^2, l^1, l^0].$$

The total mean curvature of any convex grain satisfies the relation.

$$M = 2\pi\, D_{\mathrm{Fe}}, \tag{1.2}$$

where D_{Fe} is Feret's diameter of the grain. On the basis of this relation the total mean curvature is given a very simple geometrical interpretation as a quantity proportional to the average width of the grain. The total Gaussian curvature has a constant value $C = 4\pi$, also for non-convex, but single connected grains. In the case when the grain G represents a polyhedron, M and C are sometimes called the total curvature of the edges or of the corners of a polyhedron, respectively. The quantity C is of great importance in stereology as a quantity proportional to the number of grains.

On the basis of the above characteristics the so called basic stereological parameters, referred to the unit volume of the region R, are defined:

$$\begin{aligned}
V_V &= \frac{\sum_i^N V_i}{V_R} \quad \left[\frac{l^3}{l^3}\right], \qquad & S_V &= \frac{\sum_i^N S_i}{V_R} \quad \left[\frac{l^2}{l^3}\right],\\
M_V &= \frac{\sum_i^N M_i}{V_R} \quad \left[\frac{l^1}{l^3}\right], \qquad & N_V &= \frac{N}{V_R} \quad \left[\frac{l^0}{l^3}\right].
\end{aligned} \tag{1.3}$$

Let us note that the parameters (1.3) assume values which depend exclusively on the volume of the region R and on the values of the characteristics of the set of grains $\{G_i\}$. Thus they depend neither on their location nor on their orientation with respect to this region.

In order to obtain the estimators of the parameters (1.3) in the stereological analysis a section method of three kinds: planar, linear and point method is used.

In the planar analysis, information is collected from the geometrical mosaic of the traces of grains, visible on plane sections of the region R, distributed on random, isotropically and uniformly (randomness of IUR type). The elements of information are the areas A_i, the length of the perimeters U_i and the number n_A of grain traces. The direct result of planar analysis are the parameters called stereological densities:

$$A_A = \frac{\sum_i^{n_A} A_i}{A}, \qquad U_A = \frac{\sum_i^{n_A} U_i}{A}, \qquad N_A = \frac{n_A}{A}, \tag{1.4}$$

where A represents the total area of all sections of the region R. In the case where the grains are arranged uniformly in the region R, it suffices to take into consideration one or a few sections parallel to each other.

Linear analysis consists in the measurement of the length of intercepts L_i and counting the number of intercepts n_L formed by traces of grains with the measurement lines distributed randomly (or uniformly) on plane sections.

From the linear analysis we obtain the densities:

$$L_L = \frac{\sum_i^{n_L} L_i}{L}, \qquad N_L = \frac{n_L}{L}, \tag{1.5}$$

where L denotes the total length of measured lines. The linear analysis contains less information.

The point analysis consists in a random or uniform distribution of points on plane sections of the region R. Counting comprises the total number of points n_p hitting grain traces and the total number of points P distributed on plane sections. The result of point analysis is the density

$$P_P = \frac{n_P}{P}. \tag{1.6}$$

The stereological densities (1.4), (1.5), and (1.6) represent essential elements of the estimators of the basic parameters (1.3). They have been listed in Table 1. So far several methods of their derivation have been proposed. They are important also for non-convex grains, except the estimator of the parameter M_V, which refers only to convex grains (Bodziony, 1965). The most abundant information is obtained from planar analysis. In the classical

TABLE 1. Estimation of basic stereological parameters.

Quantity	Definition	Estimation obtained from: planar analysis	linear	point
Volume fraction V_V	$\frac{\sum V_i}{V_R}$ $\left[\frac{l^3}{l^3}\right]$	A_A $\left[\frac{l^2}{l^2}\right]$	L_L $\left[\frac{l^1}{l^1}\right]$	P_P $\left[\frac{l^0}{l^0}\right]$
Specific surface S_V	$\frac{\sum S_i}{V_R}$ $\left[\frac{l^2}{l^3}\right]$	$\frac{4}{\pi}U_A$ $\left[\frac{l^1}{l^2}\right]$	$4N_L$ $\left[\frac{l^0}{l^1}\right]$	
Specific total mean curvature M_V	$\frac{\sum M_i}{V_R}$ $\left[\frac{l^1}{l^3}\right]$	$2\pi N_A$ $\left[\frac{l^0}{l^2}\right]$		
Specific number N_V	$\frac{N}{V_R}$ $\left[\frac{l^0}{l^3}\right]$			

$$A_A = \frac{\sum A_i}{A}, \quad U_A = \frac{\sum U_i}{A}, \quad N_A = \frac{n_A}{A}, \quad L_L = \frac{\sum L_i}{L}, \quad N_L = \frac{n_L}{L}, \quad P_P = \frac{n_P}{P}.$$

stereological analysis, the essence of which is the random distribution of single planes, straight lines or points, it is not possible to obtain the estimator for N_V. For the assessment of N_V a specification of the shape of grains, is necessary. At present several methods of determining the distribution of spherical grains and counting of their specific number N_V are available. A precursor of these method was Wicksell (1925).

In the present-day stereological analysis much greater possibilities of a quantitative characteristics of the sets of convex grains are based on the random distribution of coupled sections of a pair of parallel planes, a pair of parallel straight lines or a pair of points. This method has been developed by Wiencek (1996).

A complete set of stereological parameters (1.3) is a direct basis for defining – by way of creating the quotients – the four groups of secondary parameters, listed in Table 2.

TABLE 2. Secondary stereological parameters.

	V_V	S_V	M_V	N_V
V_V	1	$\frac{S_V}{V_V} = \frac{\sum S_i}{\sum V_i}$	$\frac{M_V}{V_V} = \frac{\sum M_i}{\sum V_i}$	$\frac{N_V}{V_V} = \frac{N}{\sum V_i}$
S_V	$\frac{V_V}{S_V} = \frac{\sum V_i}{\sum S_i}$	1	$\frac{M_V}{S_V} = \frac{\sum M_i}{\sum S_i}$	$\frac{N_V}{S_V} = \frac{N}{\sum S_i}$
M_V	$\frac{V_V}{M_V} = \frac{\sum V_i}{\sum M_i}$	$\frac{S_V}{M_V} = \frac{\sum S_i}{\sum M_V}$	1	$\frac{N_V}{M_V} = \frac{N}{\sum M_i}$
N_V	$\frac{V_V}{N_V} = \frac{\sum V_i}{N}$	$\frac{S_V}{N_V} = \frac{\sum S_i}{N}$	$\frac{M_V}{N_V} = \frac{\sum M_i}{N}$	1

The most often used parameters are those listed in the last, fourth line of Table 2. Their interpretation is simple. They represent the volume, the surface area and the total mean curvature for a grain of the set $\{G_i\}$ on the average.

The basic parameters, and consequently the secondary parameters have an important property: they are independent of the arrangement of grains in the region R. Extension of the definitions of these parameters to the case of a multi-component material does not present any difficulty.

In the planar stereological analysis the areas A_i and the lengths of perimeters U_i of traces of grains visible on a section of the region R are measured. Their number n_A is also counted. This makes it possible to determine the distribution and the moments of these quantities; thus, in particular, the average values:

$$\bar{A} = \frac{\sum_i^{n_A} A_i}{n_A}, \qquad \bar{U} = \frac{\sum_i^{n_A} U_i}{n_A}. \tag{1.7}$$

From the measurements and counts realized in the linear analysis it is possible to determine the distribution of the intercept lengths, as well as moments of this distribution, hence in particular the average value:

$$\bar{L} = \frac{\sum_i^{n_L} L_i}{n_L}. \tag{1.8}$$

In Section 3 it will be shown that the average values $\bar{A}$, $\bar{U}$, $\bar{L}$ can be referred directly to the characteristics of grains $\{G_i\}$.

Both the distributions of areas, perimeter lengths and intercept lengths as well as their moments depend exclusively on the set of grains $\{G_i\}$. They do not depend upon the arrangement of grains.

2. Stereological analysis of air-entrained concrete

A complete stereological analysis of air-entrained concrete comprises both the characteristics of the substructure formed by the set of pores and the substructure formed by the set of the aggregate grains. The third component – the paste – plays the role of the matrix. On the basis of performed measurements and counts it is possible to determine the estimators, listed in Table 1, separately for the pores and for the aggregate grains. Taking advantage of the fact that pores have a spherical shape, it is possible to determine their size distribution and to the calculate their specific number N_V with reference both to the unit volume of the specimen of concrete and to the unit volume of the paste. As a consequence, it is possible to determine all secondary parameters for the substructure of pores listed in Table 2.

In principle, the stereological analysis may be reduced to a quantitative assessment of the substructure of pores, provided that its aim is the evaluation of its freeze-thaw resistance.

Powers (1949) proposed to correlate the freeze-thaw resistance of concrete with the spacing between the surfaces of pores located in immediate neighbourhood of each other. He took into consideration a set of pores of the same size. From Power's considerations, however, any assumption concerning the arrangement of pores in the paste does not follow. Power's spacing factor $\bar{L}$ has the dimension of length. It is expressed by the following formulae:

$$\bar{L} = \begin{cases} \dfrac{T_p}{4N} & \text{for } \dfrac{p}{A} \leqslant 4.342, \qquad (2.1\text{a}) \\ \dfrac{3}{\alpha}\left[1.4\left(1+\dfrac{p}{A}\right)^{\frac{1}{3}} - 1\right] & \text{for } \dfrac{p}{A} > 4.342, \qquad (2.1\text{b}) \end{cases}$$

where, according to the notation used by Powers:

T_p – total length of chords in the paste,

N – number of traces of pores cut by the measuring lines,

α – specific surface of pores per unit volume of pores (and not per unit volume of the paste or concrete),

p – paste – concrete volume ratio,

A – pores – concrete volume ratio.

For $p/A = 4.342$ both formulae coincide and give equal values of $\bar{L}$, independently of α. Determination of the spacing factor $\bar{L}$ is based on stereological linear analysis.

It is possible to demonstrate that the following relationship is fulfilled

$$\frac{T_p}{4N} = \frac{p}{\alpha A}, \qquad (2.2)$$

and formulae (2.1) can be rewritten in a unified form

$$\bar{L} = \begin{cases} \dfrac{p}{\alpha A} & \text{for } \dfrac{p}{A} \leqslant 4.342, \qquad (2.3\text{a}) \\ \dfrac{3}{\alpha}\left[1.4\left(1+\dfrac{p}{A}\right)^{\frac{1}{3}} - 1\right] & \text{for } \dfrac{p}{A} > 4.342. \qquad (2.3\text{b}) \end{cases}$$

On the basis of formula (2.3a) it is possible to give the geometrical interpretation of the spacing factor $\bar{L}$. According to the definitions of the parameters A, p, α we get

$$A = \frac{V_a}{V_c}, \qquad p = \frac{V_p}{V_c}, \qquad \alpha = \frac{S_a}{V_a}, \tag{2.4}$$

where:

V_c – volume of concrete,

V_p – volume of paste,

V_a – total volume of pores,

S_a – total surface area of pores.

The formulae (2.4) and (2.3a) yield:

$$\bar{L} = \frac{\dfrac{V_p}{V_c}}{\dfrac{S_a}{V_a}\dfrac{V_a}{V_c}} = \frac{V_p}{S_a}. \tag{2.5}$$

From this formula it follows that the spacing factor $\bar{L}$ represents the volume of paste per unit surface area of pores. If each pore were surrounded by a spherical shell of paste, the thickness of this shell would be approximately equal to $\bar{L}$.

From formulae (2.3) another important conclusion follows. The spacing factor $\bar{L}$ does not characterize the arrangement of pores in the paste because it is a function of parameters independent of their location.

The European Standard EN 480-11, 1998, accepted by the Polish Normalisation Committee, presents a procedure – based also on the linear analysis – for the determination of pore size distribution (thus also of its moments) and calculation of specific number of pores N_V. This is undoubtedly an additional and very important characteristics of the substructure of pores. However, this characteristics is also independent of the arrangement of pores.

From linear stereological analysis it is possible to distinguish different arrangements of pores belonging to the same set. For this purpose the distribution of the lengths of chords in the paste have to be determined. A measure of the degree of nonhomogeneity of the arrangement may be the variance of the distribution. It should be expected that the variance will attain the smallest value in the case of homogeneous arrangement.

In the last years a great progress has been made in the technique of preparing polished sections of air-entrained concrete. This has been incorporated into the European Standard, which recommends the application of accurately described procedures for polishing the thick sections and contrasting the pore traces. The proper preparation of thick sections is a necessary condition of correct identification of traces of pores by means of automatic analysis of images, utilising the procedures of morphological transformations. Thus, the quantitative assessment of the structure of air-entrained concrete based on automatic planar stereological analysis is fully justified. Numerous studies in this field have been published recently, cf. Brzezicki (1993), Brzezicki and Kasperkiewicz (1998), Kasperkiewicz and Załocha (2000), Młynarczuk and Zając (2000a, 2000b).

3. Spatial interpretation of the average values (A, V, L) obtained in stereological analysis

Let us consider a convex grain G characterized by four basic parameters $[V, S, M, C = 4\pi]$. The common part of the grain G and its intersection by the plane E represents a convex body degenerated to a plane figure GE. Its characteristics are:

$$\begin{aligned} V(GE) &= 0, & S(GE) &= 2A(GE), \\ M(GE) &= \frac{\pi}{2} U(GE), & C(GE) &= 4\pi, \end{aligned} \tag{3.1}$$

where $A(GE)$ and $U(GE)$ are the area and the perimeter length of the plane section formed as a result of the intersection of grain G by the plane E in a position chosen with respect to the grain G.

As a result of sweeping out all the positions of plane E with respect to grain G (with $GE \neq \emptyset$) in a way according randomness (IUR), we obtain a set of plane figures. It has been found (Kendall and Moran, 1963) that the mean values of areas and perimeter lengths of plane sections are expressed by the formulae

$$\langle A(GE) \rangle = \langle A \rangle = 2\pi \frac{V}{M} \tag{3.2}$$

$$\langle U(GE) \rangle = \langle U \rangle = \frac{\pi^2}{2} \frac{S}{M} \tag{3.3}$$

Let us consider now the penetration of grain G by straight line L. The common part of grain G and straight line L is a convex body degenerated to an intercept. Its basic functionals are as follows:

$$V(GL) = 0, \quad S(GL) = 0, \quad M(GL) = \pi L, \quad C(GL) = 4\pi, \tag{3.4}$$

where L represents the length of the formed intercept GL. The mean value $\langle L(GL) \rangle$ of the intercept lengths L, determined for the set of intercepts formed for all positions of the straight line L with respect to the grain G, such that $GL = \emptyset$ is expressed by the formula

$$\langle L(GL)\rangle = \langle L\rangle = 4\frac{V}{S}. \tag{3.5}$$

The mean values $\langle A\rangle$, $\langle U\rangle$, $\langle L\rangle$ are interrelated by

$$\frac{\langle U\rangle\langle U\rangle}{\langle A\rangle} = \pi. \tag{3.6}$$

The last formula is valid for each convex grain G.

It can be demonstrated that the form of the formulae (3.2), (3.3), (3.5) and of (3.6) is preserved for analogous mean values, but defined for a set convex grains (Bodziony, 2000).

Let us consider a set of grains $\{G_i\}$, $i = 1, \ldots, N$, characterized by four parameters $\left[V_i, S_i, M_i, C_i = C = 4\pi\right]$ for $i = 1, \ldots, N$.

The mean values for i-th grain are determined by using the formulae (3.2), (3.3) and (3.5):

$$\langle A_i\rangle = 2\pi\frac{V_i}{M_i}, \qquad \langle U_i\rangle = \frac{\pi^2}{2}\frac{S_i}{M_i}, \qquad \langle L_i\rangle = 4\frac{V_i}{S_i}. \tag{3.7}$$

The mean values referring to the entire set of grains $\{G_i\}$ are defined as the weighted means of the respective mean values defined for particular grains. We obtain:

$$\langle\langle A\rangle\rangle = \frac{1}{\sum_i^N M_i}\sum_i^N M_i\langle A_i\rangle = \frac{2\pi\sum_i^N V_i}{\sum_i^N M_i} \tag{3.8}$$

$$\langle\langle U\rangle\rangle = \frac{1}{\sum_i^N M_i}\sum_i^N M_i\langle U_i\rangle = \frac{\frac{\pi^2}{2}\sum_i^N S_i}{\sum_i^N M_i} \tag{3.9}$$

$$\langle\langle L\rangle\rangle = \frac{1}{\sum_i^N S_i}\sum_i^N S_i\langle L_i\rangle = \frac{2\pi\sum_i^N V_i}{\frac{\pi^2}{2}\sum_i^N S_i} \tag{3.10}$$

We set:

$$\sum_i^N M_i = M_0, \qquad \sum_i^N S_i = S_0, \qquad \sum_i^N V_i = V_0. \tag{3.11}$$

Then we get:

$$\langle\langle A\rangle\rangle = 2\pi\frac{V_0}{M_0}, \tag{3.12}$$

$$\langle\langle U\rangle\rangle = \frac{\pi^2}{2}\frac{S_0}{M_0}, \tag{3.13}$$

$$\langle\langle L\rangle\rangle = 4\frac{V_0}{S_0}. \tag{3.14}$$

For the set of grains $\{G_i\}$ also the form of the relation between the mean values is preserved

$$\frac{\langle\langle U\rangle\rangle\,\langle\langle L\rangle\rangle}{\langle\langle A\rangle\rangle} = \pi. \tag{3.15}$$

It is also possible to prove that the average values $\bar{A}$, $\bar{U}$, $\bar{L}$ of areas, perimeter lengths and intercept lengths, defined by the formulae (1.7) and (1.8), obtained by using stereological analysis, are the estimators of the unknown mean values $\langle\langle A\rangle\rangle$, $\langle\langle U\rangle\rangle$ and $\langle\langle L\rangle\rangle$ referred to the set of grains $\{G_i\}$:

$$\langle\langle A\rangle\rangle \approx \bar{A}, \qquad \langle\langle U\rangle\rangle \approx \bar{U}, \qquad \langle\langle L\rangle\rangle \approx \bar{L}, \tag{3.16}$$

Consequently the relation

$$\frac{\bar{U}\,\bar{L}}{\bar{A}} \approx \pi \tag{3.17}$$

can be treated as a criterion of the accuracy of measurements and counts, realised by performing stereological analysis.

Let us denote by V_R, S_R the volume and surface area of specimen R containing the set of grains $\{G_i\}$, $i = 1, \ldots, N$, with their total volume and surface area V_0, S_0, respectively. It can be shown that the mean length of intercepts $\langle\langle L_m\rangle\rangle$ formed by straight lines with the matrix satisfies the relation

$$\langle\langle L_m\rangle\rangle = 4\frac{V_R - V_0}{S_R + S_0}. \tag{3.18}$$

In a particular case, when convex grains degenerate into flat figures or cracks ($V_0 = 0$), we get

$$\langle\langle L_m\rangle\rangle = 4\frac{V_R}{S_R + S_0}. \tag{3.19}$$

Then the mean value $\langle\langle L_m\rangle\rangle$ does not depend on the arrangement of cracks in the specimen R.

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