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Communications

STEREOLOGICAL METHODS IN CEMENT-BASED MATERIALS TECHNOLOGY

A survey of my research group's activities during the past half of a century

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ABSTRACT

Four topics of high engineering relevance in which we were involved are introduced herein. Aggregate packing in concrete is of obvious relevance: denser packings lead to reduced cement demands, while modern developments in the (super) high performance range of cementitious materials are based on particle packing. Fiber reinforcement efficiency in concrete that we have studied extensively offers a relatively simple stereological problem for which Cauchy laid down the fundament. In the third problem of damage analysis the dispersion of small cracks in concrete is at issue. Insight into damage characteristics would be relevant in all (fracture) mechanical experiments. This topic can equally be linked to Cauchy. In both cases, the data acquisition by stereological methods is indicated. The fourth topic is of high actual relevance. It involves porosimetry in computer-simulated (virtual) cementitious materials, ultimately aiming permeability estimation. The stereological problems involved are indicated, and - again – Cauchy can be linked to finding solutions. Finally, new, mostly yet unpublished developments are indicated aiming for more economic procedures as well as improving reliability of permeability estimates by nano-packing of globules, so that ultimately this methodology could replace the laborious and expensive (and biased) experimental route.

Keywords: Cauchy, aggregates, fibers, cracks, pores

INTRODUCTION

This paper is intended as a "farewell message" to my dear colleagues and friends in the ISS community, focusing on what I personally learned in this ISS community and showing *to which engineering problems I applied it in the concrete technology world during the past half of a century*. Hence, I herewith want to show my gratitude to the very community: it enriched my life, as a concrete technologist and as a human being!

I enjoyed my first experiences with STEREOLOGISTS in the ISS community during the Bern conference in 1971. I was involved in an extensive study in the *concrete materials field* for my PhD study that I completed in 1973. For most PhD committee members out of "my" field, the 329 pages of my dissertation were probably outside their capabilities, *and the stereological approach made this an even more serious problem*. Although I received my PhD degree – yet not without some overt animosity– for my colleagues in Delft I was considered for the rest of my active life an outsider who always knew better… As a consequence, the appointment in 1997 to professor was due to cooperation partner Beijing Jiaotong University in China, not to my home institution…

So, I figured that as a young Dr. I should convince colleagues that *stereology* was a powerful tool for solving practical engineering problems in the field of concrete technology. A highly relevant field was *steel fibre reinforced concrete* (SFRC), which presented the *simplest stereological conditions I could imagine*: dispersed lineal elements in space. This task was supposedly easier than doing so for the *damage analysis* problems I had faced earlier in my PhD study. Motivation to accept this mission was also coming from serious violations to stereological concepts I had discovered in both fields.

Looking backwards, the research efforts in the previous period involved *four major engineering topics* with stereological background. The two selected 'promotion' topics (SFRC and damage analysis) are nowadays quite established. Other contributions came from all over the world. The 'French school' can nevertheless be mentioned here because of their stereological expertise (Carcassès, Ollivier and Ringot, 1989; Dequiedt and Redon, 2001; Granju and Ringot, 1989). The *particle packing problem* is the oldest one in the series and elaborated by extensive physical experimenting as well as by computer simulation efforts, the latter facilitating for verification possibilities and assessment of peculiar effects (*e.g*., Brazil Nut Effect (BNE), see Stroeven and He, 2011c and 2012). The *permeability* issue in virtual materials is of recent date. Hence, not all our achievements could be laid down yet in major publications (Stroeven; Stroeven and Słovik; Li and Stroeven). It is a field also receiving major attention by a number of famous research institutions in the world, although studies are generally not based on DEM packing of the binder particles.

Cauchy concepts (Cauchy, 1882) constituted a solid *stereological basis* under the major part of our investigations (Li and Stroeven*; Stroeven and Słovik). Yet, additionally, the Double Random Multiple Tree Structuring (DRaMuTS) system for assessment of pore network topology should be mentioned because inspired by a typical stereology-related robotics method (LaValle and Kuffner, 2001). Also, probing pores by stars is equally inspired by a stereological method used in life sciences (Gundersen, *et al*., 1988; Stroeven). As a consequence, we can safely state that various aspects of porosimetry and permeability estimation of concrete can constitute an interesting playground for *concrete technology researchers familiar with stereological theory*!

PARTICLE DISPERSION AND PACKING

The subject matter of part of my PhD study was concrete in which the largest aggregate fraction was replaced by mono-size 16 mm ceramic (‘steatite’) balls that could easily be distinguished in sections because of their shape and colour (Fig. 1). The ‘realcrete’ was produced as 250 mm cubic concrete samples, containing different amounts of steatite. After hardening, a 25 mm disturbed boundary layer was removed by sawing. Thereupon, the resulting 200 mm cubes were serially sectioned, so that all spheres were (at least) visible in two successive sections. On an electric tablet we recorded three points on perimeters of all steatite grain sections. This allowed 3D reconstruction of the steatite structure in the specimens. The effect of steatite content on distribution parameters could be assessed in this way. Among the interesting results was the detection of the Brazil Nut Effect (BNE) in the specimens with the largest steatite content (Stroeven and Stroeven, 1999; Stroeven and He, 2011c; 2012).

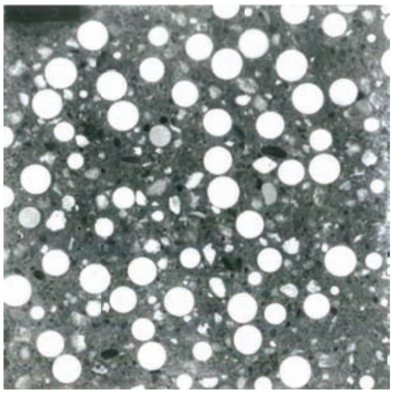

Fig. 1. Section of steatite concrete.

Fig. 2. Representative particles from fluvial gravel (top) and from crushed rock (bottom).

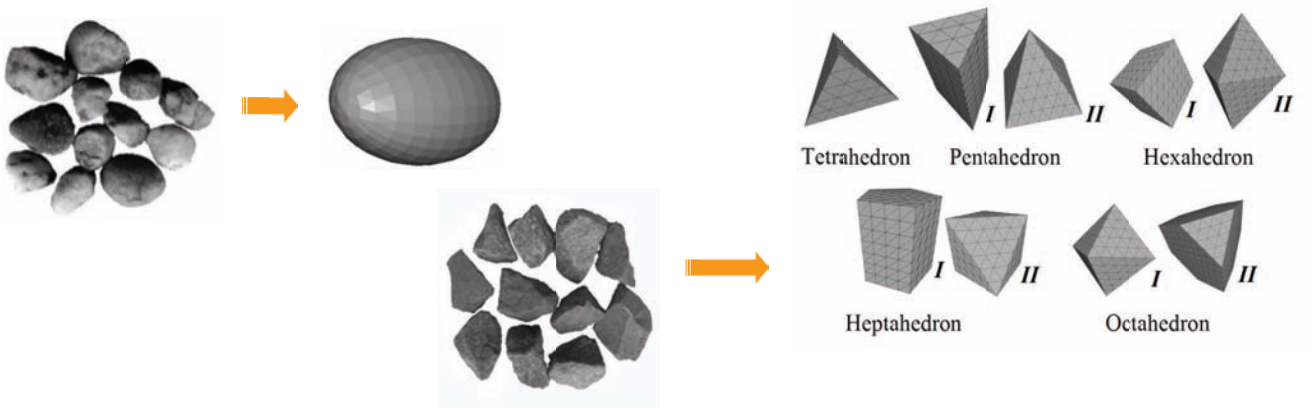

Fig. 3. Simulation strategy of arbitrary shaped aggregate; (left): differently shaped ellipsoids represent river gravel; (right): nine regular polyhedra with facet number 4~8 were selectively employed to represent river gravel aggregate in Fig. 2.

This topic was continued decades later on computer-made "steatite" concrete. In doing so, the nearest neighbour distribution of the grains could be compared with aforementioned experimental data as depicted by Fig. 4. Major deviations were found due to the inaccuracies in sawing! (Stroeven, 1973, 2015; Stroeven *et al.*, 2007; Le and Stroeven, 2012).

Simulating in cementitious materials the effects of aggregate of fluvial origin or of crushed rock on mechanical properties was also possible (He, *et al*, 2011a and 2011b; 2012). Ellipsoid-type or polyhedron-types of particles were employed for the simulation, respectively, as demonstrated by Figs. 2 and 3. Fig. 5 proves that practical mixes could be accurately simulated.

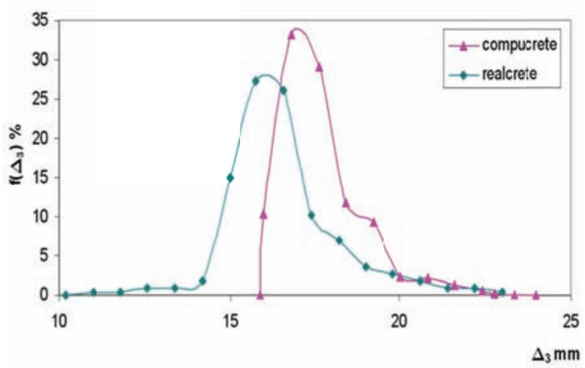

Fig. 4. Frequency distributions of the nearest neighbor, Δ_3, among 16 mm ceramic balls in 200 mm sample of "realcrete" (Fig. 1) and "compucrete" (computer-made concrete), respectively. Differences are mainly due to sawing inaccuracies for serial sectioning of the realcrete.

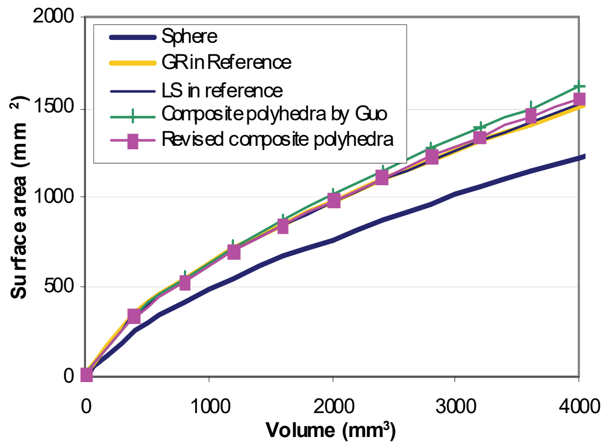

Fig. 5. S-V curves of crushed rock types (GR and LS), of composite polyhedra and of the sphere. Field data by Guo (1988) were employed as well as revised data to get an even better correspondence with reference data from an experimental approach by Erdogan, *et al.* (2006).

Of course, major upgrading of material qualities into the (super) high performance range of concrete (exceeding that of regular steel qualities) is based on *particle packing phenomena*! Purpose is to exploit physical (van der Waals) forces in an optimum way. More material developments also rely at least partly on packing of particles. However, this single example may already proof the relevance and actuality of the discussed subject.

STEEL FIBRE REINFORCED CONCRETE

Buffon formulated in 1770 a problem relevant for concrete technology. The original statement of the so called "needle problem" is given in Stroeven and Hu (2006). It could readily be transformed into the following expression by which fiber distributions in concrete can be analyzed

$$\overline{P_L} = \frac{2}{\pi} L'_A \tag{1}$$

Herein $\overline{P_L}$ is the average number of intersections of a line grid and (steel) fiber projections in an X-ray image, such as in Fig. 6. Further, L'_A is the total projected length perpendicular to the grid direction per unit of area of the image. Saltikov proposed more than 2 centuries later his directed (and random) secants analysis methodology that is making use of eq. (1).

Our first application of the Buffon/Saltikov method involved the analysis of the dispersion of different fiber types in SFRC. Two water to cement ratios by weight (*w/c*=0.4 and 0.6) and two fineness moduli of the aggregate (f=3.27 and f=3.89), were employed, maximum grain size being 8 mm. Note that the fineness modulus (FM) is an empirical figure obtained by adding the total percentages of the sample of an aggregate retained on each of a specified series of sieves, and dividing the sum by 100. A total of 48 specimens, measuring 70x200x1000 mm, were strain-controlled tested to beyond ultimate, thereupon removed from the testing machine and sectioned so that a set of about equally sized prismatic specimens resulted from each full-size specimen (Fig. 6 at the top). Visualization was by way of X-raying slices (Fig. 6, bottom) of pre-loaded concrete specimens and applying orthogonal grids. For detailed information see (Stroeven, 1979c; 2009; Stroeven and Shah, 1978; Stroeven and Dalhuisen, and Stroeven 1996; Babut, 1986; Stroeven and Słovik; Stroeven and Hu, 2007). Among other things, it provided information on the reinforcement efficiency of the different fiber types and it rendered possible to interpret phenomenological behavior of the SFRC specimens in structural terms.

The remaining prisms were subjected to 3-point bending experiments and the cubes to splitting tensile strength tests in either one of orthogonal directions. For specific information on crack development and fiber distribution parameters of the full-size specimens, see (Stroeven, 1979c; 2009; Stroeven and Shah, 1978; Stroeven and Dalhuisen, 1996; Stroeven and Babut, 1986).

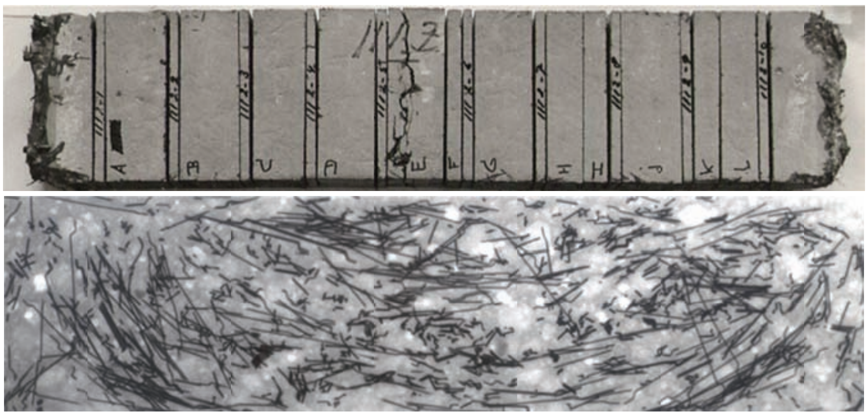

Fig. 6. (bottom) X-ray radiograph of a "vertical slice" (totaling 10 in this case) of a test-loaded (and cracked) SFRC specimen of 70x200x1000 mm (top). Compaction by gravitation was in vertical direction. Analysis is by orthogonal line grids.

The steel fiber reinforced 3-point bending experiments as well as the splitting tensile tests were provided with additional fiber dispersion characteristics by *counting fibers in orthogonal sections*. This is related to the *Cauchy* concepts (Cauchy, 1882). One of the counting planes is parallel to the fracture plane, the second perpendicular to it and parallel to the bottom of the specimen. It was found, as an example, that fiber anisometry, based on the so called Stroeven concept (in this case a mixture of 2D and 3D "randomly" oriented fibers), was directly reflected by anisotropy in splitting tensile strength at increasing volume fraction of fibers (Fig. 7) (Stroeven and Babut, 1986; Stroeven, 1993). See, *e.g.*, Stroeven (2009) for a large selection of also other relevant contributions to the field of fiber reinforcement in concrete.

Note further that in SFRC we deal with *line length in space* and in the forthcoming damage analysis approach with *surface area in space*. Cauchy provided the relationships that are at the basis of counting observations and determination of total line length (SFRC) and total surface area (Damage analysis), respectively (Cauchy, 1882).

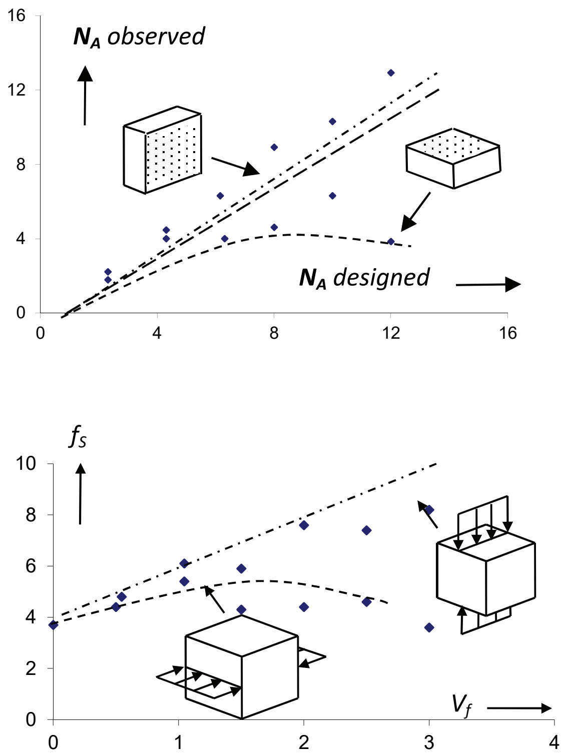

Fig. 7. Anisometry in steel fiber dispersion (top) and anisotropy of splitting tensile strength f_s in N/mm^2 of steel fiber reinforced cubes at increasing fiber volume fraction V_f (bottom).

For the fibre case of Fig. 6, upon application of the Stroeven concept of a mixture of 2D and 3D fibre portions, the following equation is obtained (Stroeven, 1979c; 2009)

$$L_V^{'} = \left[\frac{1}{2} + \left(\frac{2}{\pi} - \frac{1}{2}\right)\omega\right] L_V \qquad (2)$$

where ω is the total length ratio of the 2D fibre portion over that of the total amount of fibres. Mono-size fibers are assumed in this case, however, also for multi-size and curved fibers, similar models have been expanded. In eq. (2), L_V is the total fiber length per unit of volume and L_V' the component in the plane of the 2D portion; so, in the case of Fig. 6 oriented perpendicular to the compaction direction of the prismatic specimen. Consequently, it is the effective part of the fiber reinforcement because loading direction will be perpendicular to the plane of the X-ray image.

DAMAGE ANALYSIS

Buffon/Saltikov provided also the basis for quantitative analysis of cracks in concrete. By as well adopting the Stroeven concept for spatial crack development (combining at maximum, 1D, 2D and 3D portions), the orthogonal measurements of intersections of line grids with the cracks can be interpreted in spatial terms. Fig. 8 concerns the case of direct compression, requiring only a 3D and 1D portion, the latter having its axis of orientation coinciding with that of the loading. Hence, the solution for the spatial interpretation of cracking can be based on a section parallel to the loading direction in which the orthogonal observations $P_{LP}$and $P_{L\perp}$ suffice (Fig. 8, at the left).

In mathematical terms, it is found that the crack surface area per unit of volume, S_V, is related to orthogonal measurements in the mid-section of the compressed specimen by

$$S_V = \frac{\pi}{2}\left(P_{L\perp} + \left(\frac{4}{\pi} - 1\right) P_{LP}\right) \qquad (3)$$

In tension testing, a combination of 3D and 2D portions is required. Again, an axial section can be sampled by an orthogonal line grid. For methodological details and results of *applications,* see (Stroeven, 1973; 1979b; Stroeven and Słovik; Stroeven and He, 2011a; 2011b). Of course, any (fracture) mechanical investigation on cementitious materials can profit from the structural research efforts discussed herein, since it will promote the understanding of the recorded phenomenological behavior.

Note that these labour-intensive contributions to the aforementioned topics had to be terminated at the end of last century, because physical experimenting became too expensive. Therefore, I figured we should instead use modern computer facilities. So, the last subject is *hot,* actual and highly relevant. My PhD student and son, Martijn, developed the first *professional* Discrete Element Method (DEM) - SPACE - in our group that became operational around the millennium break (Stroeven, M., 1999). During the next years, a series of my PhD students have used it (and an upgraded version, HADES) in studies on *virtual cementitious materials (compucrete).* Particle packing problems (as discussed earlier) and particularly *permeability estimation* were the issues of interest and engineering relevance.

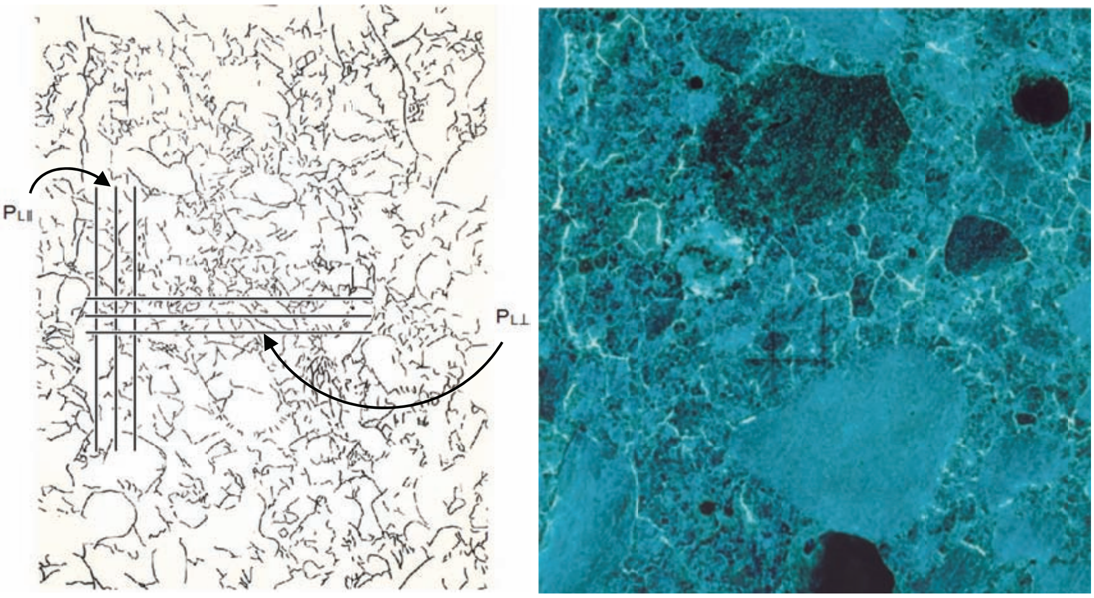

Fig. 8 (right) Part of the section image of a pre-loaded compressed prismatic concrete specimen reveals myriads of small cracks visible under UV light upon application of a fluorescent spray. (left) Manually copied crack pattern of a complete, centrally located axial section (note aggregate grain sections). Orthogonal line grids are superimposed for intersection counting.

In Stroeven and Słowik we have explicitly elaborated on the Cauchy concepts. However, for stereological readers this is "as easy as falling off a log", hence, it is avoided in this survey. Instead, the showing up of the Cauchy constants of 1/2 and $2/\pi$ in geometric averaging operations was demonstrated. This more closely reflects my *Aha Erlebnis* when I was starting to employ his concepts in our engineering fields!

POROSITY AND PERMEABILITY

Applications of the Cauchy concepts for 3D and 2D problems is not limited to the structural analysis of fibre reinforcement or crack structures. More recently they found their way in porosimetry and predictive models for permeability (Li and Stroeven 2017a,b; Stroeven, *et al*., 2015). For that purpose, our virtual materials are produced by Discrete Element Method (DEM) – despite Random Sequential Addition (RSA) methods are more frequently used in concrete technology, leading however to biased particle dispersion characteristics. The next step is hydration simulation by XIPKM (eXtended Integrated Particle Kinetics Model) that is based on spherical cement particles yielding also spherical hydrated grains (as a result of the vector-based approach), apart from inter-particle interferences (Navi and Pignat, 1996; Pignat, Navi and Scrivener, 2005) (Fig. 9).

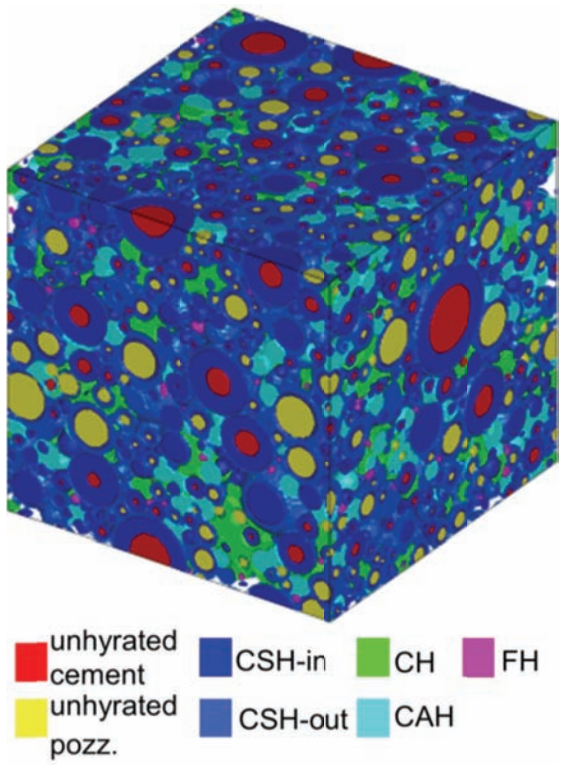

Fig. 9. Simulated hydrated blended cement paste

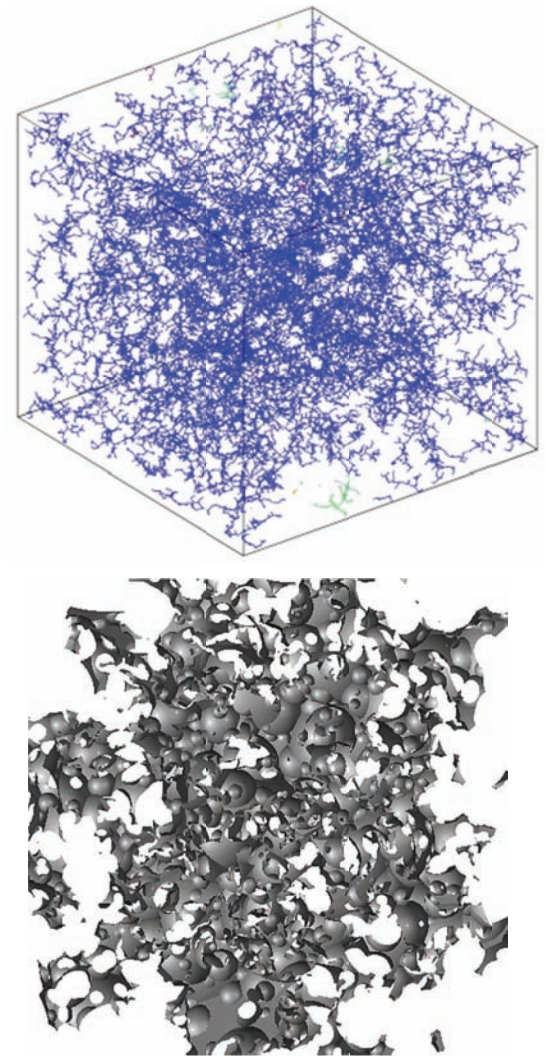

Fig. 10. (top) All pores in 90 days hydrated cement paste with w/c = 0.4 and Blaine surface area of 300 m^2/kg. Porosity is 19%. (bottom) Tortuous and complex pore structure in the vector-based hydrated system after removal of the hydrate structure (Stroeven, M., 1999).

Pore topology of the capillary network is assessed by DRaMuTS (Double Random Multiple Tree Structuring), see Fig. 10, an approach inspired by developments in robotics (as stated in the Introduction) and pore size is determined by Star Volume Measuring (SVM), inspired by a methods used in life sciences (see also the Introduction).

These investigations are based on $2\text{x}10^5$ "randomly" dispersed points in 100 μm cubes. The smoothed trees from DRaMuTS and the pore sizes by SVM are input data for a classical network analysis, finally yielding information on permeability (Stroeven and Li, 2017; Stroeven *et al.*, 2015; Li *et al.*, 2017). The complexity of the pore structure is visualized in Fig. 10.

New developments focus on the *economy of the approach*. Instead of assessing 2x 10^5 throat sizes for the network analysis, a similar number of IUR oriented pore sections are used. This avoids probing by 2-D stars in about 200 pore sections at each nodal point to find the pore throat (=smallest among pore sections in the same point) (Stroeven). It is found that the size distribution functions of throats and random sections are intimately related. Specifically, the median pore areas in the two distributions differ by the Cauchy constant ½ (Stroeven and Słovik). A second economization effort is to predict permeability from tortuosity information. In Stroeven and Słovik the linear tortuosity index of pores, R_l, is approximately expressed by

$$R_1 \approx 1 + \frac{\pi}{2}(1 - p) = \frac{l}{l'} \qquad (4)$$

Herein p is porosity and l and l' are pore length and projected pore length on a straight line. So far, simulated data based on the second half of Eq. (4) seem quite well predicted by porosity data according to the first part of it. For estimating transport characteristics of the pore system by this so called hybrid approach, we additionally require information on the *number of pore channels* and their *median 2D size*, plus a reduction factor expressing the actual conductance of the *non-spherically shaped pore sections*. The latter is determined by FET (Le, 2015; Li *et al.*, 2017). Significant reductions in efforts are expected (a journal paper is in preparation).

FUTURE RESEARCH

The last two decades we have been conducting research in the field of concrete permeability (Li, *et al.*, 2017), as aforementioned. As a follow up of the PhD study of K. Li (Li and Stroeven, 2019), this has finally resulted in nano-level explorations in the outer hydration layer of cement particles. This is the layer that causes the expansion of the hydrating particles. We have replaced this outer hydration layer of the smooth spherical surface of hydrated cement particles by packed nano-globules. In the literature, the size of such nodules is estimated as 5 nm (Jennings, 2000; 2008). From the surface of the so called inner hydration layer they start clustering in fractal-like "arms", leading to a declining global density over the outer layer (Fig. 11).

The traditional XIPKM vector approach to hydration simulation is based on spherical cement grains that expand due to hydration in a spherical way (accounting for interferences). This is shown in a 2-D low-density set up in Fig. 11 (top). The nano-approach obviously reduces pore size (and changes shape) while pore surface is roughened in Fig. 11 (bottom). This will reduce the conductance of water through a pore section. When transferring the information of *virtual cement to concrete*, we deal with additional effects due to dilution and tortuosity. It seems that the nano-approach in combination with these material parameters can bring the simulated and experimental data on concrete permeability on close distance (Li and Stroeven). Hence, instead of laborious and thus expensive testing (with accepted shortcomings and uncertainties as to the degree of water saturation of the specimens), the sketched strategy, based on virtual specimens, would not only compete favorably as to time and money savings but would be at least of equal reliability!

Problems arise in the nano-approach as to assessing size and shape changes in 3D during growth of the quasi-fractal "arms" in Fig. 11. *An interesting topic of stereological nature suitable for a next PhD study!*

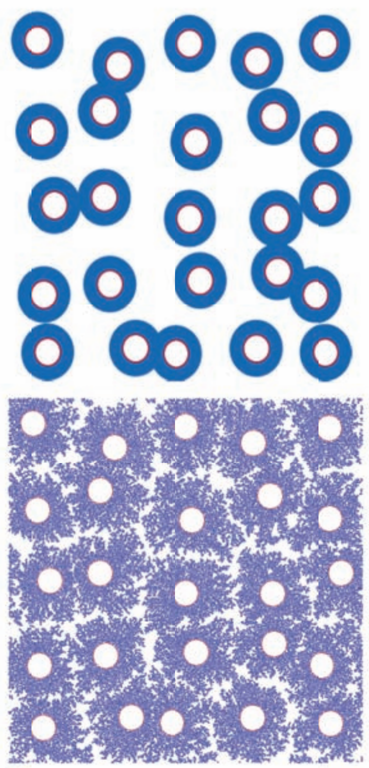

Fig. 11. The simplified structure of cement paste in 2D consisting of DEM-packed mono-size cylindrical disks under two different growth mechanisms: (top) homogenous expansion by XIPKM; (bottom) fibrous growth among mono-size globules. Amount of C-S-H in outer layer is similar in the two cases.

CONCLUSIONS

Hence, my "stereological life" started when I plunged with full ambition into extensive physical experimenting in the framework of my PhD study (*i.e.* on the very laborious *particle packing* and *damage analysis* problems in concrete) and thereupon with foreign counter-parts on *SFRC*. It resulted over the many years in a couple of hundred papers in conference proceedings and in journals in the SFRC field (of the many thousands available worldwide), *with the ambition to show stereology a very practical and reliable tool for solving engineering problems*. My last PhD student on this topic was Shui Zhonghe from Wuhan University of Technology who received his PhD in 2001 (Shui, 2001). Of course, damage analysis in concrete is intimately related in stereological terms to the fiber topic (due to the two Cauchy concepts of determining the length of a line or area of a surface, both in 3D space, by averaging of their total projections).

Studies on all topics - as already mentioned for SFRC - have resulted in series of publications in leading journals such as in IAS (Stroeven, *et al*., *2012*; Li, *et al*., 2015; Hu and Stroeven, 2006). Some publications, among others with Kai Li, are still in *statu nascendi*. Brand new developments can hereby be mentioned, such as simulating the outer hydration layer of hardened cement paste consisting of nano-size globules dispersed in fractal-like "arms" (Fig. 11); real stuff for stereologists! Yet, recently, we have given prime attention to economic developments in permeability estimation using – again – Cauchy's 3-D concept, as mentioned in this paper (see also: Stroeven; Stroeven and Słovik).

It should finally be stressed that the mentioned stereology-related topics were not the only ones that kept me busy all those years. At least, one other topic should be mentioned here, because it may offer nuance to the impression of "hard" technology activities that probably rose from the discussed topics. This one was concerned with *sustainability aspects* in concrete technology "*avant la letter*". Application focussed on a rural project in Sri Lanka with Architectural student participation, while laboratory research was conducted in the framework of cooperation with Tanzania, Brazil and particularly with Vietnam. The latter was supported by the Dutch Government (1982-2002), although political changes in the country resulted in our case (as the last accepted project) in significant budget cuts. Major objective was to reduce CO_2 emissions by partly replacing Portland cement by fine-grained rice husk ash (Bui, 2001) or non-commercial metakaolin (Vu, 2002). Mind that this was significantly before such CO_2 emissions were internationally declared leading to global warming, hence it was hard in our case to find moral and, particularly, financial support. Note that the given examples are *particle packing-driven*. This illustrates again the actual relevance of the first topic discussed herein!

At the final end of this paper it should be mentioned that *promotion of stereology in my field was also explicitly in my mind during the many years that I participated in the ISS community as "regional representative" and as Editorial Board member for Acta Stereologica and Image Analysis and Stereology.*

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