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THE QUANTITATIVE CHARACTERISTICS OF THE MICROSTRUCTURE OF BUILDING COMPOSITES

KVANTITATIVNE KARAKTERISTIKE MIKROSTRUKTURE GRAĐEVINSKIH KOMPOZITA

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Preliminary communication

Abstract: *The article deals with the algorithm of the building material structure based on its photomicrographs by means of point images statistical analysis. The obtained values allow the defining of the basic characteristics of the material structure.*

Keywords: *building materials, image processing, Nih Image, point images, statistical geometry*

Prethodno priopćenje

Sažetak: *U radu se razmatra algoritam strukture građevinskog materijala na temelju njegove fotomikrografije pomoću statističke analize točkastih slika. Dobivene vrijednosti omogućavaju definiranje osnovnih karakteristika strukture materijala.*

Ključne riječi: *građevinski materijali, obrada slika, Nih Image, točkaste slike, statistička geometrija*

1. INTRODUCTION

One of the directions of materials science that has significantly developed in the last decade is a structure-based research of the modeling and optimization of building composites [1,2]. The success of this research area is largely due to the possibility of a more effective control of the desired performance formation based on structural information, the evolving theory of the self-organization of dispersed systems [3] and the overall technical level of the improvement in the field of the research of composite materials. The qualitative results about the self-organizing processes in the materials and their models have been obtained in a number of studies [4]. A transition to the quantitative values and their correlations is more productive in choosing the optimal control actions on the composite properties by using the statistical procedures. In relation to that, it is necessary to analyze the possibility of obtaining such characteristics by simple means, by considering their content component and the possibility of building the “structure-properties” of statistical models. Such models, in comparison to the traditional “composition-properties” for material science models, have a number of advantages, i.e. a clear quantitative and qualitative interpretation, the possibility of considering the received dependences of the materials’ broad classes, the possibility of their use to express the estimation of properties, and an easy transition to the physical models.

In composite materials, the interaction between their components – binder, filler and additives, is complex [5]. This, in particular, can appear in the tendency to form the distributions of components’ particles, as well as the pores and internal separation boundary in accordance with the laws that differ from the purely random ones, given by the Poisson law. At each fixed scale level, such a deviation can belong to two types (Fig. 1).

If the attraction force between the particles dominates, they form clusters. At the opposite tendency, quasi-regular structures are formed based on the maximum repulsion principle. This distribution is easy to acquire by giving the same charges to the movable particles.

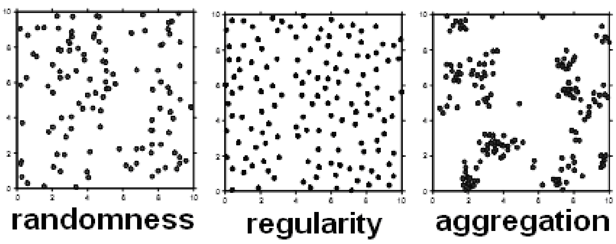

Figure 1. The nature of point distribution on a plane.

As a "reference point" for the complexity degree, it is possible to accept the DLVO theory that takes into account the only van-der-Waals and coulomb interactions. Particles obeying this theory are already characterized by structure organization and the complex interactions, i.e. periodic colloidal structures [5], long and short coagulation contacts are formed. The transition to the particles of complex nature, the possibility of structural and mechanical barriers formation, and other causes significantly complicate the accurate examination of the particles interaction in the composite binder test.

2. MAIN SECTION

One way to solve the problems arising in the study of the composite particles interaction is the transition to a simplified system - the original model. The distribution of macroscopic particles on the surface and in the bulk liquid phase [4] can be used particularly in such a model. In such systems, despite their relative simplicity and the ease of the experimental methods, processes of structure formation occur. Due to the capillary forces between particulates (2 to 5 mm), there is a preferential attraction; and what should additionally be taken into account at close range is their electrostatic, van der Waals and other types of interactions. The considered method of the processes of physical modeling in composites by using a particulate was used [4], and the introduction of a quantitative description increases its productivity.

One of the easiest ways to study the macrostructure of composites and their models based on the optical methods of studying at a relatively low increase (10x-1000x), for which a microscopic investigation in the reflected light is used which has not lost its relevance in spite of the emergence and development of more advanced techniques [6]. Enhanced with the corresponding means of fixing images (CCD-camera and the associated equipment, together with the software), the conventional microscopic study is transformed into computer microscopy techniques.

For a variety of materials, the structures observed by using the optical methods are characterized as small particles, pores, and other objects, and their spatial distribution can be examined.

Consider the possible options of the point objects of this type:

1. The particle components of the composite – the research is released if they are painted in different colors or vary in the gray scale.
2. The pores – there is no need for a stereological reconstruction of the observed object porous structure with the point approach.
3. Projections of cracks and internal borders' phase boundary on the chips or grinding – the point approach is possible to use in such objects only after the machine image segmentation of these lines and surfaces.
4. Mineral growths processed with a special treatment (e.g. phenolphthalein for lime in the cement stone, tannin for cracks).

The process of the investigation of these structures is as follows:

1. With the help of an electronic microscope eyepiece, converted to macro mode webcams and similar equipment, micrographs of a thin section or a smooth flat cleavage of the material are obtained. Further, it will be assumed that a preparation produced by appropriate means if necessary (such as a cement stone processed by a phenolphthalein developer). As an example, consider the implementation of the algorithm processing the micrographs of a plastering material based on gypsum and perlite [7] (Fig.2).

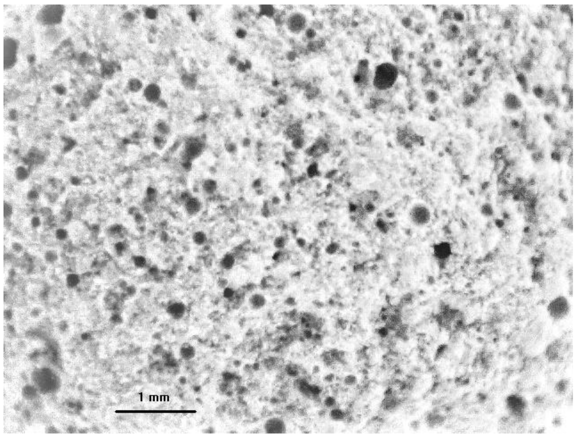

Figure 2. A photomicrograph of a material by subtracting the background light level (Scale mark - 1mm)

2. Further processing may be carried out by using a free software such as Nih Image, ImageJ and its derivatives, Image Tools and many commercial ones (e.g. Optimas). The Nih Image 1.62 Fat software [8], which has become a classic in the field of medicine and biology, was used in the emulator Executor 2.0 environment. The macro batch file for micrograph processing was written. After a background subtraction, a threshold separation image (Threshold) was carried out. Next, the filtering, which allows to remove the noise image produced by unrelated pixels was conducted. Furthermore, after the transfer to a binary image, the ultimate point of erosion (ultimate eroded point) was built. After the threshold separation and binarization, small clusters of pixels appear on the screen corresponding to the most central area of the original objects (Fig. 3).

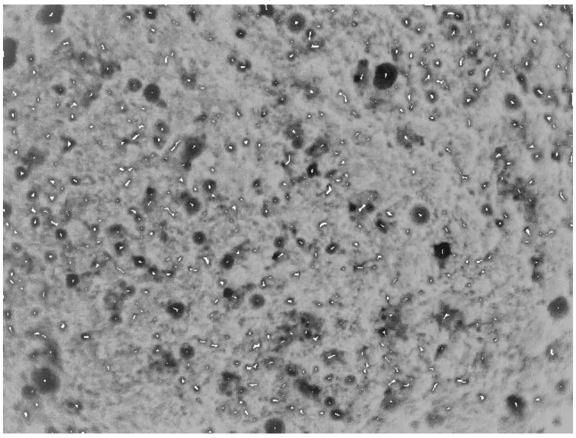

Figure 3. The ultimate erosion points imposed on the original image

Such objects may be analyzed automatically and display the values of their origin (their other parameters in the considered approach is not required) to the file (Fig. 4).

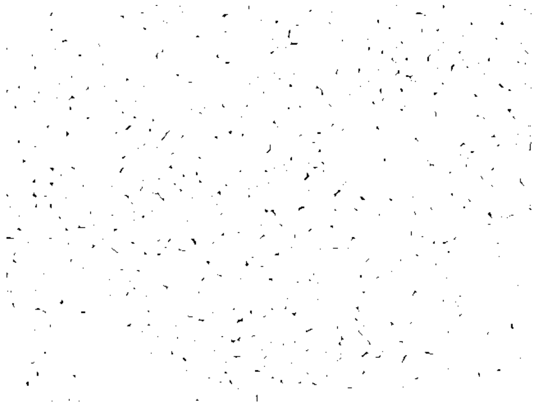

Figure 4. The ultimate erosion points of which the coordinates are determined

The third phase of the structure analysis is carried out by using the points' location on the plane and in the space analysis software (PPA (DOS), Past – Paleontological software, Ppa (mac) and others. The latter is activated as accessible on the same emulator. Consider the results of its work; they are displayed graphically.

The analysis of the distribution point can be made by a square grid (squares analysis). The square grid with side *a* (1) is imposed on the dot pattern.

$$a=\sqrt{\frac{\text{Preparation area}}{2\times\text{Number of points}}} \tag{1}$$

Figure 5 shows an example of such a construction:

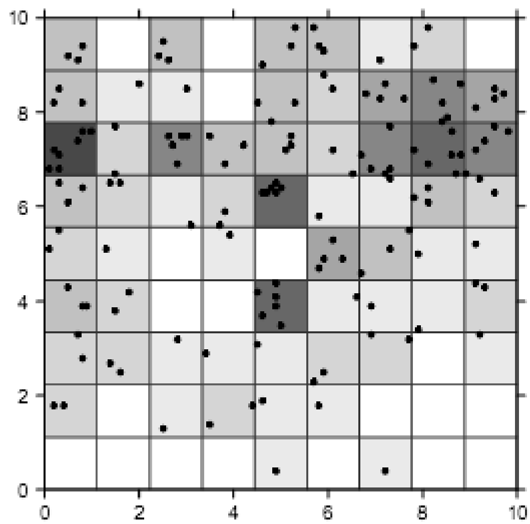

Figure 5. An analysis of the statistical characteristics of a point image by the square method

Furthermore, the number of events (in this case - of the particles or pores) in each square is calculated and the considered frequencies are processed statistically (Fig. 6).

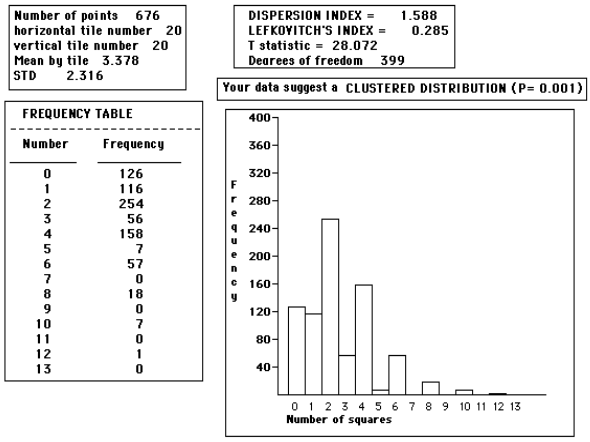

Figure 6. The results of the statistical analysis by the squares method

The essential data to decision making can be the output of the analysis results (Fig.7)

1) General data

Number of points 676

Density 18 cells/mm2

2) Indices		Random	Meaning Regular	Clustered
Dispersion Index	2.389	=1	<1	>1
Lefkovith's Index	0.495	=0	-1	+1
Packing Factor	0.067	=0	+1	
Eberdhart Index	1.185	1.27	<1.27	>1.27
Means/STD Index	2.323	close to 0	>0	
Equilateral Index	----	close to 1	close to 0	

Figure 7. The results and the boundary values of the point image statistical analysis

The dispersion index [9] or Fano factor is defined as the ratio of the variance σ^2 to the mean frequency μ (2)

$$D=\frac{\sigma^2}{\mu} \tag{2}$$

If D = 1, the image is random. This means that the set data has no dominant trend for clustering or dispersion. If D < 1, the point image has a regular structure, i.e. the point spread in the observed region is more or less regular. The dispersion index depends on the values of the mean values, which is why an additional criterion, the Lefkovitch index is used [10] (3) (in radians):

$$\varDelta=\frac{4}{\pi}\operatorname{arctg}\frac{s^2}{\sigma^2}-1 \tag{3}$$

Here s^2 - the variation of the frequency in this sample, σ^2- the variation of random distributions, the Poisson distribution is substituted for the value of the average, $\sigma^2=m$. As the dispersion index, $\varDelta$ = 0 corresponds to a random distribution, $\varDelta = -1$ - regular and $\varDelta$ = 1- cluster.

The Ebernharda Index [11] (4) also allows on the test the assessment of the grouping of the object degree, S – the standard deviation and $\bar{x}$ the average evaluation of the distance from a random point to the test.

$$I_E=\left(\frac{S}{\bar{x}}\right)^2+1 \tag{4}$$

The corresponding limit values are shown in Fig. 7. All major statistical characteristics indicate a high probability of a cluster formation in this sample.

The nature of the particles' mutual arrangement can be detected from the distribution diagram of the distance between the particles (Fig. 8). What can be found on the values of the density maxima are the approximate location of the coordination spheres (approximately 100 and 150 microns) and the mean value of the effective radius [12], characterizing the size of the "dead" space around the object under the study.

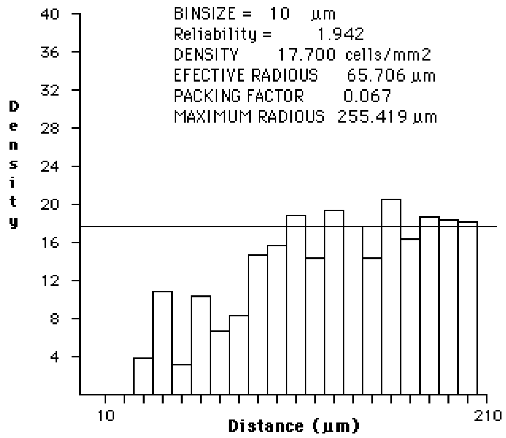

Figure 8. The distribution of distance between the particles and derivative values.

3. CONCLUSION

Thus, a consistent application of computer image and statistical processing of the micrographs and similar material structure images reveal the characteristic structure of the material and specify the conditions of their non-random distribution. This, in turn, may be interpreted as the evidence of the material self-organization process of the spatial structure that occurs at different time stages. The processes of space-time self-organization of composite materials are a significant influencing factor determining their performance.

4. REFERENCES

[1] Bentz, D. P. Three-dimensional computer simulation of Portland cement hydration and microstructure development. J. of the American Ceramic Society, 80, 1997, 3-21

[2] Bentz, D. P. Modelling cement microstructure: Pixels, particles, and property prediction. Materials and Structures, 32, 1999, 187-195

[3] Bobryshev, A. H.; Kozomazov, V. N.; Babin, L. O.; Solomatov, V. I. Sinergetika kompozitnyh materialov. Lipeck, NPO ORIUS, 1994. 152 p.

[4] Vyrovoj, V. N.; Dovgan', I. V.; Semenova, S. V. Osobennosti strukturoobrazovanija i formirovanija svojstv polimernyh kompozitnyh materialov, Odessa, TJeS, 2004, 168 p.

[5] Frolov Ju.G. Kurs kolloidnoj himii, M.,Himija, 1982, 400 p.

[6] Sarid, D. Exploring scanning probe microscopy with "Mathematica", John Wiley & Sons, Inc., New York, 1997, 262 p.

[7] Kersh, V.; Kolesnikov, A.; Lyashenko, T.; Pidkapka, M. Structurally-oriented design of the heat insulation plastering material, Proc. Of International conference of Advanced HVAC & Natural Gas Technology, May 6-9, 2015, Riga, Latvia, 4p.

[8] Schneider, C. A.; Rasband, W. S.; Eliceiri, K.W. NIH Image to ImageJ: 25 years of image analysis. Nature Methods 9, 671-675, 2012.

[9] Hoel, P. G. On Indices of Dispersion. Annals of Mathematical Statistics 14 (2): (1943), 155-162.

[10] Duncan, K. W. A modification to Lefkovitch's index of spatial distribution, Proceedings of the New Zealand Ecological Society, 1972, v. 19, 158-162 pp.

[11] Krebs, C. J. Ecological Methodology, 1999, 2nd ed., Addison-Welsey Educational Publishers, Inc., Menlo Park, 620 pp.

[12] Rodiek, R. W. The density recovery profile: A method for the analysis of points in the plane applicable to retinal studies, Visual Neuroscience, 1995, v. 6, 95-111 pp.

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