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BIMODAL-STRUCTURED ANISOTROPIC MATERIAL: GRAIN SIZE ESTIMATION

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ABSTRACT
The study directs towards the estimation of grain size in materials with anisotropic bimodal-structured morphology. Model material was prepared by compression moulding from plastics pellets with known bimodal size distribution. The estimation was based on the routine approach using planar and linear sections. Results from both the suggested novel method and the traditional method proposed by Standard ASTM E 112 were compared with the known values of grain size. The new method was assessed as a precise tool for grain size estimation even in such a complex morphology.
Keywords: anisotropic grain size estimation, Voronoi tessellation, ASTM E 112

1. INTRODUCTION

1.1. Grain size estimation in isotropic structure

The grain structure, more precisely, its grain size, can be described by basic characteristic which is, in the 3D context, the mean grain volume $\mathbf{E}v$ ($\mathbf{E}v=1/N_V$, N_V is the number of grains per unit volume) or the mean grain width $\mathbf{E}w$ (the mean caliper or Feret diameter). These quantities are inaccessible by a direct measurement, the 2D and 1D approaches prevail and the “size” is represented by the mean planar profile area $\mathbf{E}a$ ($\mathbf{E}a=1/N_A$, N_A is the mean number of profiles per unit area) or by the mean intercept length $\mathbf{E}L$ ($\mathbf{E}L=1/N_L$, N_L is the mean number of grain intercepts per unit length of the test line). Ordinarily, the recommendations of the Standard ASTM E 112 [1] or similar EN ISO 643 [2] are used for an estimation of the mean grain volume from planar or linear sections. General stereological relations between N_V, N_A and N_L can be written as follows [3], [4]:

$$N_V = c'(N_A)^{3/2}, \quad N_V = c''(N_L)^3, \quad N_A = c(N_L)^2. \tag{1}$$

The dimensionless scale invariant factors c, c' and c'' depend on the type of grain structure. For isotropic materials, the ASTM Standard assumes universal values c=0.788, c’=0.80 and c”=0.566. However, in general the factors c, c' and c'' depend on the structure characteristics, namely

$$c' = \sqrt{\frac{\mathbf{E}v}{(\mathbf{E}w)^3}}\ , \ c'' = \frac{\mathbf{E}v^2}{(\mathbf{E}s/4)^3}\ , \ c = \frac{\mathbf{E}w\mathbf{E}v}{(\mathbf{E}s/4)^2} = \left(\frac{c''}{c'}\right)^{2/3}, \tag{2}$$

where $\mathbf{E}w$ is the mean calliper diameter and $\mathbf{E}s$ is the mean cell surface.

1.2. Anisotropic modification

In the case of linear-planar anisotropic grain system, the plane and line sections in the main directions significantly differ and the grain size estimation is more difficult. Quantities N_{Lx}, N_{Ly}, N_{Lz} – numbers of grain intercepts per unit length parallel to x, y, z axes – should be measured. Similarly, quantities N_{Ax}, N_{Ay}, N_{Az} characterize the numbers of profiles per unit area perpendicular to the x, y, z-axes. Then the corresponding mean intercept lengths $\mathbf{E}L_\bullet=1/N_{L\bullet}$ and the mean profile areas $\mathbf{E}a_\bullet=1/N_{A\bullet}$ can be approximately evaluated. Standard ASTM E 112 recommends estimating the mean cell volume by the formula

$$1/\mathbf{E}v = N_V = 0.566\ N_{Lx}N_{Ly}N_{Lz}. \tag{3}$$

A novel approach to the grain size estimation suggested in this paper is based on an idea that it is possible to convert a homogeneous strongly anisotropic tessellation to an “equiaxial” one by a simple transformation. Firstly, let us to define the ratios $t_y=N_{Ly}/N_{Lx}$ and $t_z=N_{Lz}/N_{Lx}$. Conversion to the equiaxial tessellation can be achieved by the elongation of the anisotropic tessellation t_y-times in y direction and t_z-times in z direction. Formula (1a) estimates the grain size from the planar sections (note $N_{Axt} = N_{Ayt} = N_{Azt}$):

$$N_V = N_{Vt}t_yt_z = c'(N_{Axt})^{3/2}t_yt_z = c'(N_{Axt}\ N_{Ayt}\ N_{Azt}\ (t_yt_z)^2)^{1/2} = c'(N_{Axt}t_yt_z\ N_{Ayt}t_z\ N_{Azt}t_y)^{1/2}.$$

Hence

$$N_V = c'(N_{Ax}N_{Ay}N_{Az})^{1/2}. \tag{4}$$

Similarly, formula (1b) estimates grain size from linear sections (note $N_{Lxt} = N_{Lyt} = N_{Lzt}$):

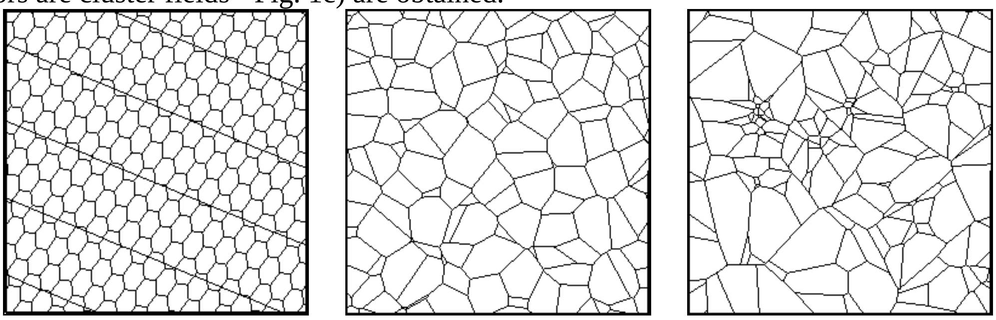
$$N_V = c''N_{Lx}\ N_{Ly}\ N_{Lz}\ . \tag{5}$$

It is evident from comparison of the formulae (4) and (5) with (1a), (1b) that the same constants c', c'' are used in the relations between the estimates of N_V obtained by profile or intercept counts but that the arithmetic means (relating to all possible sections) occurring in (1a), (2b) are replaced by the geometric means of estimates obtained in three suitably oriented mutually perpendicular section planes or lines. The same constant c occurring in equation (1c) also relates $(N_{Ax}N_{Ay}N_{Az})^{1/3}$ and $(N_{Lx}N_{Ly}N_{Lz})^{2/3}$.

1.3. Voronoi tessellations

A tessellation is the space filling system of cells (grains). The standard Voronoi tessellation is the result of simultaneous isotropic radial growth with constant rate from point nuclei (germs) arbitrarily arranged in the space. The growth is locally stopped whenever adjacent grains come into contact. Voronoi tessellations are good models of polycrystalline grain structures or cellular tissues.

Properties of the Voronoi tessellation are defined by the spatial distribution of points (generators) of the generating point process. By changing its type, tessellations with a narrow (generators are point lattices and displaced point lattices - Fig. 1a), medium (Poisson Voronoi tessellation – PVT - generators are distributed uniformly at random - Fig. 1b) and broad distribution of cell sizes (generators are cluster fields - Fig. 1c) are obtained.



a) b) c)

Figure 1: Planar sections of three different types of Voronoi tessellations. Grain size distribution extends from left to right.

A spatial tessellation generates in its 2D and 1D sections the induced planar or linear tessellations. Only such induced tessellations are available for an examination in the case of real opaque materials and the properties of the original spatial tessellation must be estimated by means of suitable stereological formulas. However, all parameters of computer simulated tessellations can be determined with an arbitrary accuracy. Then it is possible to look for a simulated structure with similar properties of sections and to expect that also the relations between the induced and spatial structures will be similar.

1.4. *w-s* diagram

It follows from equations (1and 2) that the important size characteristics influencing the relations between induced and spatial tessellations are the mean caliper diameter $\mathbf{E}w$ and the mean cell surface

Es. They determine the intensities of induced Voronoi tessellations and, consequently, are the most natural parameters characterizing and classifying any spatial tessellation. For model tessellations, they can be found with an arbitrary accuracy by computer simulation.
This is the basic idea of the *w-s* diagram (Fig. 2), which is a graphical representation of the proposed classification. It was originally introduced in [3] as a useful tool for the grain size estimation from planar and line sections. In the *w-s* diagram, any unit (i.e. **E**v=1) tessellation is represented by the point {**E**w, **E**s} in the $\{w,s\}$ plane and the position of this point directly determines also the values of the c, c' and c'' parameters used in equations (1).
Other characteristics of the examined tessellations (shape factors, quantiles, and, in particular, coefficients of variation CV v, CV v', CV v'' of the cell volume, profile area and chord length, resp.) are evaluated simultaneously and can be plotted as labels (marks) in selected points.
Various *w-s* diagrams based on computer simulations are presented on http://fyzika.ft.utb.cz/voronoi/ws/ws.htm. The central part of the *w-s* diagram is shown in Fig. 2. Tessellations generated by displaced lattices (simple cubic – c, cubic body centred - bcc and face centred - fcc), Johnson-Mehl model (JM) and Neyman-Scott cluster fields (PG for Poisson globular fields, PS for Poisson spherical fields). HEX denotes the tessellations by regular hexagonal prisms (upper branch describes plates, the lower one rods) and PVT denotes the Poisson-Voronoi tessellation.

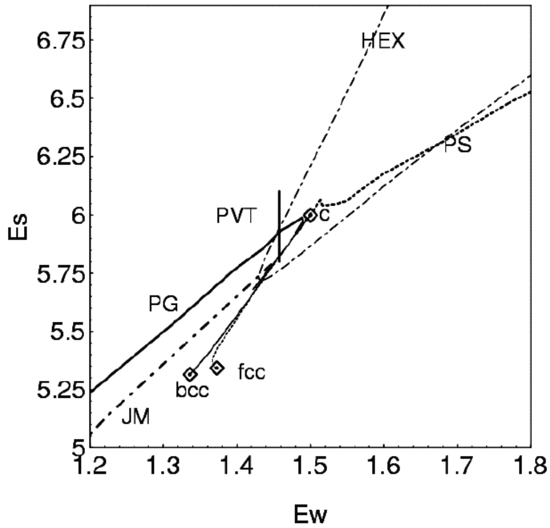


Fig. 2: Central part of w-s diagram.

2. EXPERIMENTAL

2.1. Material

Compression-moulded PVC pellets were used as a real anisotropic material suitable for examination. The mean pellets size was measured as 49 mm^3. This material is formed by two basic components – paste-forming PVC and plasticizer. The pellet surfaces were covered by carbon paste to improve the recognition of pellet boundaries. Prepared blend was isothermally annealed and compression-moulded in the cylindrical mould using a manual press. From the resulting cylindrical moulding a rectangular prism specimen was cut (Fig. 3). Specimen sides were either perpendicular or parallel to the direction of deformation. Finally, the specimen surface was polished and scanned by a computer scanner. Obtained images of the surface structure were used as the base for the following analysis.

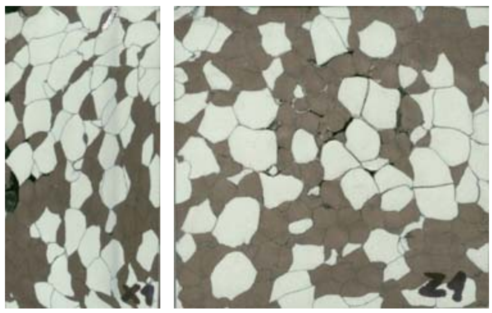


Figure 3: Surface of the sample

Let xyz be a coordinate system, axes x and y are horizontal and axis z is vertical. The compression was vertical, parallel to the axis z (Fig. 3). Thus, horizontal surfaces z_1 and z_2 of the specimen were perpendicular to the compression direction, whereas surfaces y_1, y_2 (perpendicular to y) and x_1, x_2 (perpendicular to x) were parallel with the compression direction.

3. RESULTS AND DISCUSSION

3.1. Profile area measurement

Images of sample sides were magnified 3×, a rectangular region of suitable area was selected and the number of profiles inside of this region was counted. The Gundersen frame [5] was used for the edge correction. Table 1 display the results of this analysis.

face perpen-dicular to	profile area a [mm^2]	CV a	N_A [mm^{-2}]
x	9.86	0.82	0.101
y	9.14	0.73	0.109
z	15.89	0.85	0.063

line direction	int. length L [mm]	CV L	N_L
x	3.25	0.636	0.307
y	3.34	0.647	0.299
z	1.87	0.646	0.534

Now we must estimate the proper *c'* and *c"* values. Using equation (1) we are able to compute from $N_{A\bullet}$ and $N_{L\bullet}$ corresponding *c* value *c* = 0.66. We have to find a tessellation with known *c* value (representing line in *w-s* diagram) and CV *a* near 0.8 [6]. In Fig. 4 is part of w-s diagram. It is obvious that proper parameters are *c'* = 0.54 and *c"* = 0.29. Our structure is composed by grains of two types. The same are properties of tessellations generated by globular clusters – there are small cells near cluster centre and bigger external cells. Indeed our point is on curve for globular clusters.

Using formula (1a) and values of $N_{A\bullet}$ from table, the number of grains per unit volume is 0.014 mm^{-3} (corresponding grain volume is 70 mm^3 (formula (1b) and $N_{L\bullet}$ return the same value – *c'* and *c"* are connected by *c*). These values are in good agreement with known cell volume 49 mm^3.

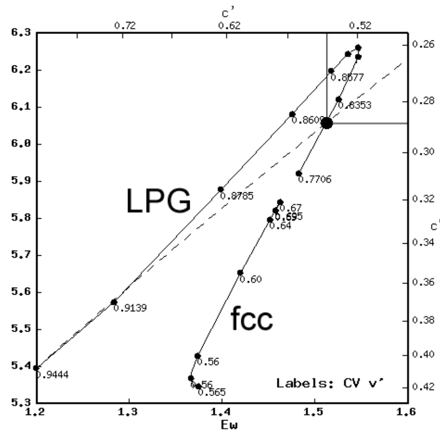


Figure 4: Part of w-s diagram. Dashed line corespond c=0.66. LPG is globular clusters placed in simple cubic lattice, fcc is displaced face-centered cubic lattice. Numbers at points denotes CV a. Disc is point in w-s diagram proper to experimental structure.

4. ACKNOWLEDGEMENTS

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