Anisotropic Grain Size Estimation Using Computer Simulations

PETR PONÍŽIL[1], JAROSLAV PROCHÁZKA[1], ROMAN ČERMÁK[1] AND IVAN SAXL[2]

[1]Tomas Bata University in Zlín, Faculty of Technology, TGM 275, 762 72 Zlín,
[2]Mathematical Institute, Acad. Sci. of Czech Rep. and Faculty of Mathematics and Physics,
[2]Dept. of Probability and Mathematical Statistics, Charles University, Praha,
CZECH REPUBLIC,
http://fyzika.ft.utb.cz/voronoi/

Abstract: - A method for the grain size estimation of an anisotropic polycrystalline material from planar and linear sections in the main anisotropy directions is suggested. The derived formulae were tested on an anisotropic material represented by compression-moulded pellets. Properties of its planar and linear sections were known and also the grain volume was measured for comparison with computed values.

Key-Words: - grain size estimation, anisotropic material, tessellation, computer simulations

1 Introduction

1.1 Isotropic structure

The basic characteristic of the grain structure is its grain size, which means, in the 3D context, the mean grain volume $\mathbf{E}v$ ($\mathbf{E}v=1/N_V$, N_V is number of grains per unit volume) or the mean grain width $\mathbf{E}w$ (the mean calliper or Feret diameter). As 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]:

$$N_V = c'(N_A)^{3/2}, \qquad (1)$$

$$N_V = c''(N_L)^3, \qquad (2)$$

$$N_A = c\,(N_L)^2. \qquad (3)$$

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

$$c' = \sqrt{\frac{\mathbf{E}v}{(\mathbf{E}w)^3}}\,, \qquad (4)$$

$$c'' = \frac{\mathbf{E}v^2}{(\mathbf{E}s/4)^3}\,, \qquad (5)$$

$$c = \frac{\mathbf{E}w\mathbf{E}v}{(\mathbf{E}s/4)^2} = \left(\frac{c''}{c'}\right)^{2/3}, \qquad (6)$$

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

1.2 Anisotropic extension

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

$$1/\mathbf{E}v = N_V = 0.566\, N_{Lx}N_{Ly}N_{Lz}. \qquad (7)$$

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. Using this transformation, the mean intercept length parallel to the x-axis, $\mathbf{E}L_{xt}$, remains the same, $\mathbf{E}L_{xt}=\mathbf{E}L_x$ ($N_{Lxt}=N_{Lx}$), whereas the mean intercept lengths in other directions change: $\mathbf{E}L_{yt}=t_y\mathbf{E}L_y$ ($N_{Lyt}=N_{Ly}/t_y$) and $\mathbf{E}L_{zt}=t_z\mathbf{E}L_z$ ($N_{Lzt}=N_{Lz}/t_z$). For the mean profile areas after transformation holds $\mathbf{E}a_{xt}=t_yt_z\mathbf{E}a_x$, $\mathbf{E}a_{yt}=t_z\mathbf{E}a_y$ and $\mathbf{E}a_{zt}=t_y\mathbf{E}a_z$. Then for the corresponding numbers of profiles per unit area can be written: $N_{Axt}= N_{Ax}/(t_yt_z)$, $N_{Ayt}= N_{Ay}/t_z$ and $N_{Azt}= N_{Az}/t_y$. The mean grain volume changes by this transformation as well: $\mathbf{E}v_t=t_yt_z\mathbf{E}v$ and for number of grains per unit volume can be written $N_{Vt}=N_V/(t_yt_z)$. After this procedure we obtain an equiaxial tessellation and can use formulas (1-3) for the transformed quantities. Formula (1) 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

$$N_V = c'(N_{Ax}N_{Ay}N_{Az})^{1/2}. \qquad (8)$$

Similarly, formula (2) estimates grain size from linear sections (note $N_{Lxt} =N_{Lyt} =N_{Lzt}$):

$$N_V = N_{Vt}t_yt_z = c''(N_{Lxt})^3t_yt_z= c''N_{Lxt}\, N_{Lyt}t_y\, N_{Lzt}t_z\ .$$

Hence

$$N_V = c''N_{Lx}\, N_{Ly}\, N_{Lz}\ . \qquad (9)$$

It is evident from comparison of the formulae (8) and (9) with (1), (2) 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 (1), (2) 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 (3) 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 and displaced point lattices), medium (Poisson Voronoi tessellation – PVT - generators are distributed uniformly at random) and broad distribution of cell sizes (generators are cluster fields) are obtained.

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 a real opaque material 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 (1-6) that the important size characteristics influencing the relations between induced and spatial tessellations are the mean calliper diameter $\mathbf{E}w$ and the mean cell surface $\mathbf{E}s$. They determine the intensities of induced Voronoi tessellations and, consequently, are the most natural parameters characterising 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. 1), 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. $\mathbf{E}v$=1) tessellation is represented by the point $\{\mathbf{E}w, \mathbf{E}s\}$ 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 (1-3).

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 the Internet page http://fyzika.ft.utb.cz/voronoi/ws/ws.htm.

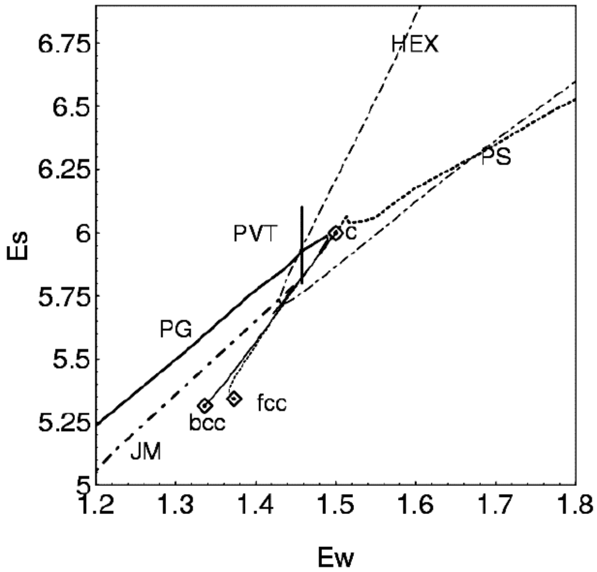

Fig 1.: Central part of the w-s diagram: tessellations generated by displaced lattices (simple cubic – c, cubic body centred - bcc and face centred - fcc), Johnson-Mehl model (JM) and Neyman-Scott cluster fields (PG for Poisson globular fields, PS for Poisson spherical fields). HEX denotes the tessellations by regular hexagonal prisms (upper branch describes plates, the lower one rods) and PVT denotes the Poisson-Voronoi tessellation.

2 Experimental

2.1. Material

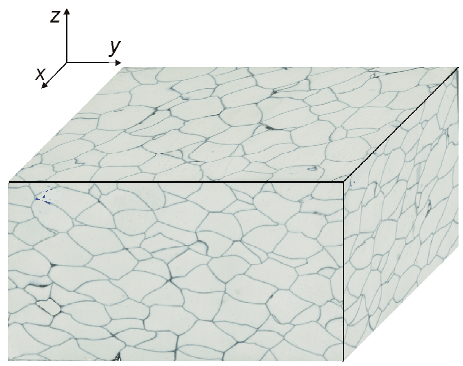

Fig. 2.: Sample system of coordinates

Compression-moulded pellets were used as a real anisotropic material suitable for examination. Plasticized PVC pellets were manufactured by Aliachem j.c., Subsidiary Fatra. This material is formed by two basic components – paste-forming PVC and plasticizer. In order to improve the recognition of pellet boundaries in the final product, the pellet surfaces were covered by carbon paste in the concentration of 1 wt. %. Prepared blend was isothermally annealed at 170°C for 1 hour and subsequently compression-moulded in the cylindrical mould using a manual press. From the resulting cylindrical moulding with diameter 10.5 cm and height 6 cm, a rectangular prism specimen of the size 5.0×3.4×2.7 cm^3 was cut (fig. 2). 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.

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. 2). Thus, horizontal surfaces z_1 and z_2 (see Figure 3- z_2 is the central face) 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 (see Figure 3).

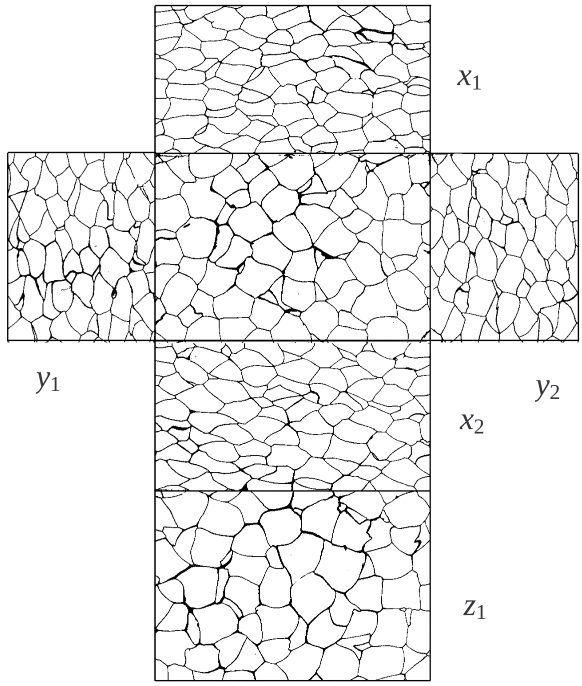

Fig. 3:Unfolded surface of sample B.

2.2 Grain volume measurement

In order to determine the volume distribution of pellets, weights of randomly chosen 147 items were measured. Pellet volumes were then estimated from these weights using the known specific gravity of 1.40 g/cm^3. As shown in Figure 4, the corresponding volume distribution is rather narrow; the true mean pellet size is 133 mm^3 with the standard deviation of 16 mm^3. Pellets are incompressible.

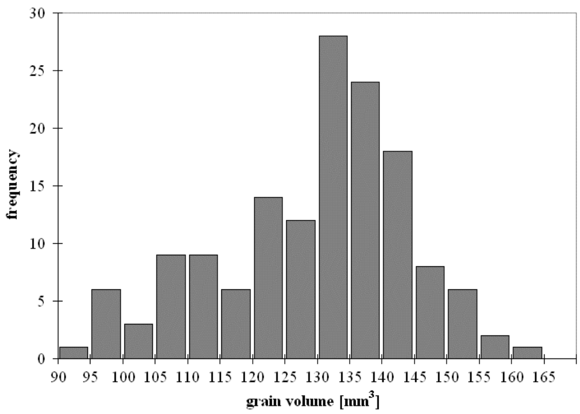

Fig. 4:. The histogram of the grain volumes

2.3 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. Tables 1 and 2 display the results of this analysis.

Table 1. Mean profile areas in all examined sample faces

face	area [mm^2]	total number of profiles	mean profile area **E**a [mm^2]
sample A			
z_1	1344	50	26.9
z_2	1344	37	36.3
y_1	556	33	16.8
y_2	556	31	17.9
x_1	611	39	15.7
x_2	611	44	13.9
sample B			
z_1	1156	47	24.6
z_2	1156	39	29.6
y_1	533	34	15.7
y_2	533	34	15.7
x_1	867	52	16.7
x_2	867	57	15.2

The geometric means of estimated $N_{A\bullet}$'s collected in table 2 are 0.050 and 0.053 mm^{-2} for the samples A and B, respectively. The corresponding mean profile areas are 19.9 and 18.9 mm^2, respectively.

Table 2. Mean profile areas in section planes

plane perpendicular to	mean profile area **E**$a_\bullet$ [mm^2]	number of profiles per unit area $N_{A\bullet}$ [mm^{-2}]
sample A		
x	14.8	0.068
y	17.3	0.058
z	31.6	0.032
sample B		
x	15.7	0.064
y	15.9	0.063
z	27.1	0.037

2.4. Intercept length measurement

In surface images, grids with a line distance of 3.3 mm (in the sample unit, in 3× magnified image it was 1 cm) were drawn. The number of intercepts for each line was counted. Results are summarized in Tables 3 and 4.

Table 3. Mean intercept lengths

test line direc-tion	face	total line length [mm]	total number of inter-cepts	inter-cept length **E**$L_\bullet$ [mm]	numbers of inter-cepts per unit length $N_{L\bullet}$ [mm^{-1}]
sample A					
x	z_1	450	98	4.59	0.218
x	z_2	444	89	4.99	0.200
x	y_1	200	47	4.26	0.235
x	y_2	250	58	4.31	0.232
y	z_1	444	101	4.40	0.227
y	z_2	438	91	4.81	0.208
y	x_1	250	63	3.97	0.252
y	x_2	200	46	4.35	0.230
z	y_1	228	98	2.33	0.429
z	y_2	243	97	2.51	0.398
z	x_1	240	89	2.70	0.370
z	x_2	234	98	2.39	0.418
sample B					
x	z_1	317	78	4.06	0.246
x	z_2	317	66	4.42	0.226
x	y_1	146	42	3.47	0.288
x	y_2	146	37	3.94	0.254
y	z_1	315	77.5	4.06	0.246
y	z_2	320	71	4.50	0.222
y	x_1	229	55	4.17	0.240
y	x_2	229	50	4.58	0.218
z	y_1	149	59	2.53	0.395
z	y_2	149	61	2.45	0.409
z	x_1	213	85	2.52	0.398
z	x_2	213	91	2.34	0.427

Table 4. Mean total intercept lengths in different directions

direction parallel to	intercept length $\mathbf{E}L_{\bullet}$ [mm]	numbers of intercepts per unit length $N_{\bullet}$ [mm^{-1}]
sample A		
x	4.54	0.220
y	4.38	0.228
z	2.48	0.403
sample B		
x	3.97	0.252
y	4.33	0.231
z	2.46	0.407

The geometric means of estimated $N_{L\bullet}$'s are 0.272 and 0.287 mm^{-1} for the samples A and B, respectively. The corresponding mean chord lengths are 3.67 and 3.48 mm, respectively.

2.5. Image analysis

Surfaces of sample B were digitalised and processed by a image analysis program. The area of each profile and length of each chord were computed. Knowing these numbers we are able to estimate other characteristics of structure - CV *a* and CV *L*.

Table 5. The variation of profile areas in different section planes

plane perpendicular to	coefficient of variation of profile area CV $a_{\bullet}$
sample B	
x	0.57
y	0.51
z	0.60

Table 6. The variation of intercept lengths in different directions

direction parallel to	coefficient of variation of intercept lengths CV $L_{\bullet}$
sample B	
x	0.51
y	0.56
z	0.53

Values of CVs for Poisson-Voronoi tessellation are CV *a* = 0.69 and CV *L*=0.58. Our values are less due to to the fact that the distribution of grain volumes is narrow and the sections have special orientations with respect to the grain anisotropy.

3. Results & Discussion

Now we must estimate proper *c'* and *c''* values. Using equation (3) we are able to compute corresponding *c* values, namely 0.675 for the sample A and 0.642 for the sample B.

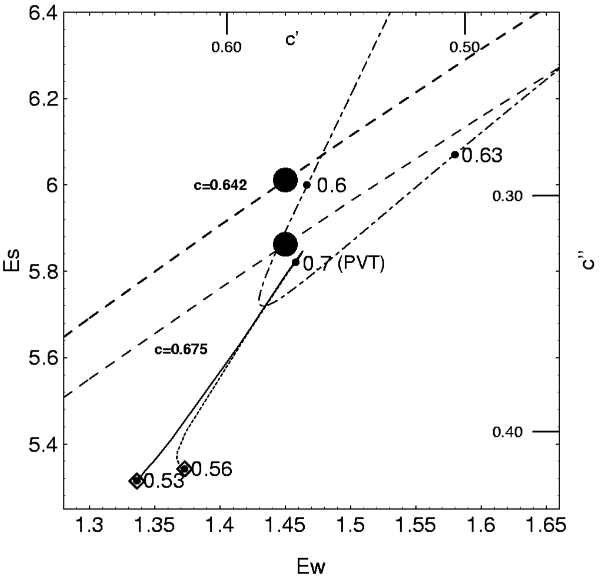

*Fig. 5: Part of the w-s diagram. Lines denote tessellations based on different point processes (only tessellations generated by displaced bcc and fcc lattices and those ones formed by hexagonal prisms are plotted – see fig. 1). Line labels denotes the values of CV a. Top dashed resp. bottom line corresponds to c = 0.675 (sample A - bottom line) and c = 0.642 (sample B - top line). The circles indicate points in the w-s diagram approximately representing our experimental structure (the estimated value of CV a and the fact the compressed pellets are plates are taken into account). The point corresponding to the values recommended by ASTM Standards lies outside of this diagram at the position {**E**w=1.16, **E**s=4.84}.*

Then the estimates **E***w*=1.45 for the both samples and **E***s*=5.86 (sample A) resp. 6.01 (sample B) are obtained. The corresponding *c'* and *c''* values (using equations (4) and (5) and **E***v*=1) are *c'* = 0.572 and *c''* =0.317 and 0.294 for the samples A and B, respectively.

3.1. Grain size estimation based on profile counts

Formula (8) suggests the method for grain size estimation from planar sections by characteristic planes of the grain structure. The necessary estimates of $N_{A\bullet}$ are available in the Table 2. Results for different *c'* (ASTM or *w*-*s* based) computed from equation (8) are presented in the table 7.

Table 7. Grain size estimated from profile counts by different methods

method	*c'*	N_V [mm^{-3}]	**E***v* [mm^3]
sample A			
ASTM	0.80	0.0090	111
w-s	0.586	0.0066	154
sample B			
ASTM	0.80	0.0098	102
w-s	0.586	0.0072	141

3.2 Grain size estimation based on intercept counts

The ASTM Standards suggests the formula (7) for the estimation of the grain size from the intercept count. It is also possible to use the formula (9) with the values of *c''* estimated on the basis of the *w-s* diagram. The necessary values of $N_{L\bullet}$ are available in the Table 4.

Table 8. Grain size estimated from intercept counts by different methods

method	*c''*	N_V [mm^{-3}]	**E***v* [mm^3]
sample A			
ASTM	0.566	0.0114	87
w-s	0.317	0.0064	154
sample B			
ASTM	0.566	0.0134	75
w-s	0.294	0.0070	141

4. Conclusions

The true mean grain volume as measured directly was 133 [mm^3], hence N_V is 0.0075 [mm^{-3}]. The constants *c'*, *c''* proposed by the ASTM underestimates **E***v* (overestimates N_V), whereas the behaviour of estimates based on the *w-s* diagram is just opposite. By considering the mean values of the estimates of N_V as obtained for the both samples A and B, the following results are obtained: the ASTM approach overestimates N_V by 25% (profile count) and 65% (intercept count), the method based on the *w-s* diagram underestimates N_V by 8% (profile count) and 12% (intercept count) only.

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