# THE SECOND-ORDER ANALYSIS OF PARTICLE SIZES BY A TEST QUADRAT METHOD

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### ABSTRACT

For a set of convex particles on the plane the particle widths (sizes)  $h_1, h_2$  measured in two perpendicular directions form a two-dimensional random variable  $(h_1, h_2)$ . The coresponding second order moment  $E(h_1h_2)$  of  $(h_1, h_2)$  characterizes the particle size and shape. The moment  $E(h_1h_2)$  is related to the expected value  $EN_K(A)$  of the number of counted particles which are totally within a test quadrat of area A. An experimental example of application for a Fe<sub>3</sub>C-dispersion in steel is given.

Keywords: convex particle, shape, testing by quadrat, size correlation.

#### INTRODUCTION

Test quadrats are often used in the image analysis practice (Wiencek and Hougardy, 1987; Rys, 1989). There are also some theoretical investigations in this field (Saltykow, 1974; Miles, 1978).

One of the methods of analysing a two-dimensional set of randomly distributed convex particles by a test quadrat K consists on counting those particles which are totally contained within the quadrat K (and excluding the particles which intersect the border of K), Fig.1.

For a randomly positioned test quadrat K (of area A) the number of particles within K,  $N_{K}(A)$ , is a discrete random variable and its expected value,  $EN_{K}(A)$ , is a function of some distributional characteristics of the set of particles. This paper aims to discuss theoretical properties of this function and possible applications.

# WIENCEK K ET AL: TEST QUADRAT METHOD



# THEORETICAL CONSIDERATIONS

A collection  $\Omega$  of two-dimensional convex, non overlapping particles is randomly distributed on the plane  $\mathbb{R}^2$  forming there an homogeneous and isotropic random set. In the plane a Cartesian  $(x_1,x_2)$  coordinate system is given. The particles can be characterized relatively to the  $(x_1,x_2)$  coordinate system as follows. With each particle  $\omega(k) \in \Omega$  a rectangle of side lengths  $h_1(k)$  and  $h_2(k)$  (the so-called particle widths) is conjugated, where the sides are tangents to the

particle and are parallel to the  $x_1, x_2$  axes, Fig.2 (k=1, 2, ...).



Fig.2. A convex particle and the conjugated rectangle.

The center x(k) of the rectangle is taken as that of the particle. The set of particles  $\Omega$  may be represented by the set of the conjugated rectangles.

The set of particles  $\Omega$  can be described by an homogeneous marked point process (Stoyan et al., 1987)  $\Psi_2 = \{[x(k), h_1(k), h_2(k)]\}$ . Here x(k) is the center of the k-th particle, and the marks  $h_1(k)$ ,  $h_2(k)$  denote its widths. The x(k) form a simple point process in the plane  $\mathbb{R}^2$  with the point density  $N_A$ . The particle widths form a two-dimensional random variable  $(h_1, h_2)$  with fundamental moments  $E(h_1)$ ,  $E(h_2)$  and  $E(h_1h_2)$ . For the process  $\Psi_2$  the expected number EN(A) of points x(k) in a test field K of area A is equal  $A \cdot N_A$ . Because of the homogeneity and isotropy assumption the variable  $(h_1, h_2)$  is independent of the way in which the coordinate system  $(x_1, x_2)$  on the plane has been chosen. This implies the equality  $E(h_1) = E(h_2) \equiv E(h)$  where E(h) is the so-called mean caliper diameter. These moments determine the co-variance  $C_{12} = E(h_1h_2) - E^2(h)$ .

In the following analysis it is assumed that the variables  $h_1$  and  $h_2$  are discrete with values:  $h_{1i}, h_{2j}$  for  $i=1,\ldots,k$  and  $j=1,\ldots,l$ . The test quadrat K of sides of length L which are parallel to the  $x_1, x_2$  axes (L ) ( $h_{1k}$  and  $h_{2l}$ )) is positioned on the plane  $R^2$  containing the set of particles  $\Omega$ . Let  $\Omega(i,j) \in \Omega$  denotes the subset of  $\Omega$  consisting of the particles  $\omega(i,j)$  of widths  $h_{1i}$  and  $h_{2j}$  which appear with the density  $N_A(i,j)$ . A particle  $\omega(i,j)$  belong to the interior of K



For a random position of K (realized by a random translation) on the plane  $\mathbf{R}^2$  the number  $N_{K}(i,j|A)$  of particles of  $\Omega(i,j)$  being totally within K is a discrete random variable with the expected value

$$EN_{K}(i,j|A) = N_{A}(i,j)(L-h_{1i})(L-h_{2i}).$$
(1)

(2)

Then the expected value  $\text{EN}_{K}(A)$  for the  $\Omega$  particles set is

$$EN_{K}(A) = \sum_{i,j} N_{A}(i,j)(L-h_{1i})(L-h_{2j})$$

and expansion of the right side of (2) gives

$$\mathbf{EN}_{K}(\mathbf{A}) = \mathbf{L}^{2} \sum_{\mathbf{i}, \mathbf{j}} \mathbf{N}_{A}(\mathbf{i}, \mathbf{j}) - \mathbf{L} \sum_{\mathbf{i}, \mathbf{j}} \mathbf{h}_{1\mathbf{i}} \mathbf{N}_{A}(\mathbf{i}, \mathbf{j}) - \mathbf{L} \sum_{\mathbf{i}, \mathbf{j}} \mathbf{h}_{2\mathbf{j}} \mathbf{N}_{A}(\mathbf{i}, \mathbf{j}) + \sum_{\mathbf{i}, \mathbf{j}} \mathbf{h}_{1\mathbf{i}} \mathbf{h}_{2\mathbf{j}} \mathbf{N}_{A}(\mathbf{i}, \mathbf{j}).$$

Taking into account the stereological equation

$$N_{L} = N_{A} E(h) = \sum_{i,j} h_{1i} N_{A}(i,j) = \sum_{i,j} h_{2j} N_{A}(i,j)$$

and the relationships

$$N_A = \sum_{i,j} N_A(i,j)$$

and

$$N_{A}E(h_{1}h_{2}) = \sum_{i,j} h_{1i}h_{2j}N_{A}(i,j)$$

yields

$$EN_{K}(A) = L^{2}N_{A} [1 + E(h_{1}h_{2})/L^{2}] - 2LN_{L},$$
 (3)

Here  ${\rm N}_{\rm L}$  is the density of the intersections of particles with a test line.

Equation (3) indicates that  $EN_{K}(A)$  is a function of the two stereological parameters  $N_{A}$  and  $N_{L}$  as well as of the moment  $E(h_{1}h_{2})$ , it is true also for a general distribution of  $(h_{1},h_{2})$  (not only for the discrete case). Of course, the expected number EN(A) of particle centers and the expected

number EN(L) of intersections (with a test line of length L) are equal to  $L^2N_A$ and  $LN_L$  respectively. Thus equation (3) can be expressed as

$$EN_{K}(A) = EN(A)[1 + E(h_{1}h_{2})/L^{2}] - 2EN(L)$$
 (4)

Finally the moment  $E(h_1h_2)$  can be expressed by the covariance  $C_{12}$ :

$$C_{12} = EN_{K}(A)/EN(A) + 2\lambda + \lambda^{2} - 1$$
 (5)

with  $\lambda = E(h)/L$ .

Equation (5) shows that the covariance of the variable  $(h_1,h_2)$  is determined by the ratio of the expected number of particles belonging totally to

the interior of K and of particle centers in K.

For circular particles with diameters d, the covariance  $C_{12}$  is equal to the diameter variance  $\sigma^2(d)$ , which thus can be expressed by equation (5). With increasing L the value  $E(h_1h_2)/L^2$  in (3) decreases to zero rapidly and equation (3) could be used for estimating  $N_A$  by two countings: that of particles being totally within the quadrat K and that of particles intersecting two parallel borderlines of K. The two-countings method is easier than direct estimation based on counting of particle centers due to the difficulties of identification of centers.

#### EXPERIMENTAL EXAMPLE

A coagulated Fe<sub>3</sub>C-dispersion in a steel sample was investigated in order to get some information on particle size and shape. For this purpose the moment  $E(h_1h_2)$  was estimated using the test quadrat method. The estimation was performed by using of the following estimator  $\langle h_1h_2 \rangle$  based on equation (4)

$${h_1 h_2}/{L^2} = [\bar{N}_K(A) + 2\bar{N}(L)]/\bar{N}(A) - 1$$
 (6)

 $\overline{N}(A)$ ,  $\overline{N}_{K}(A)$  and  $\overline{N}(L)$  are estimators of EN(A), EN<sub>K</sub>(A) and EN(L) obtained from various test quadrats in different positions by averaging numbers. For comparison the value  $\langle h_1 h_2 \rangle$  was also established by direct measurement

of  $h_1$  and  $h_2$  for the particles. The micrographs (magnification 2000 times) were made of a polished and etched by picral specimen of carbon steel (Fe-0.6%C) (hardened and annealed in vacuum at 700°C for 600 hours). Fig. 4 presents the microstructure of the Fe<sub>3</sub>C-dispersion.





The metallographic analysis indicates that the Fe<sub>3</sub>C-particle sections are approximately convex and randomly distributed in the ferrite matrix. Thus the marked point process assumed above is an acceptable model for the Fe<sub>3</sub>C-dispersion. The means  $\overline{N}(A)$ ,  $\overline{N}_{K}(A)$  and  $\overline{N}(L)$  for the test quadrat K with side length L=20 mm (scale of micrograph at 2000 times of magnification)

were determined. The measurements on micrographs were performed by a measurement quadrat  $K_0$  with  $L_0$ =80 mm divided into 16 test quadrats K with L=20 mm. The quadrat  $K_0$  determines a square net containing 16 test-quadrats K and 10 segments with length  $L_0$ . For a randomly positioned quadrat  $K_0$  the particle centers in  $K_0$  as well as the particles being totally within the quadrats K and the intersections of the particles with the segments of length  $L_0$  were counted. (One has to note that for a particular measurement quadrat  $K_0$  the test quadrats K and the segment with length L are arranged systematically. Nevertheless for homogeneous and isotropic dispersions the estimation of the means  $\overline{N}_K(A)$  and  $\overline{N}(L)$  by systematic testing is equivalent to the estimation by a random testing procedure.)

The microstructure was analysed by using of 43 randomly positioned disjoint quadrats  $K_0$ . The whole number of particles was 1529, 925 particles were totally in the interior of the 688 test quadrats K while 871 particles were intersected by the 430 segments of length  $L_0$ . The means together with the corresponding standard deviations s(  $\cdot$ ) are equal to:

$$\begin{split} &\bar{\mathrm{N}}(\mathrm{A}) \ = \ 2.222, \qquad \mathrm{s}(\bar{\mathrm{N}}(\mathrm{A})) \ = \ 0.049 \\ &\bar{\mathrm{N}}_{\mathrm{K}}(\mathrm{A}) \ = \ 1.344, \qquad \mathrm{s}(\bar{\mathrm{N}}_{\mathrm{K}}(\mathrm{A})) \ = \ 0.041 \\ &\bar{\mathrm{N}}(\mathrm{L}) \ = \ 0.506, \qquad \mathrm{s}(\bar{\mathrm{N}}(\mathrm{L})) \ = \ 0.015 \end{split}$$

The obtained values are presented at the scale of the micrographs (magnification 2000 times). Substitution of these values into equation (6) gives  $\langle h_1 h_2 \rangle = 24.120 \text{ mm}^2$ . The accuracy of the  $E(h_1 h_2)$  estimation is determined by the precision of  $\bar{N}(A)$ ,  $\bar{N}_K(A)$  and  $\bar{N}(L)$ . The total differential method applied to eq. (6) yields a value of 13.10 mm<sup>2</sup> for the standard deviation of  $\langle h_1 h_2 \rangle$ .

Next,  $\rm h_1$  and  $\rm h_2$  values for two perpendicular directions (parallel to  $\rm x_1$  and  $\rm x_2$  axes) were measured for all 1212 particles in 22 randomly chosen sam-

pling areas of the microstructure. For the obtained pairs  $(h_1, h_2)$  the two-dimensional empirical distribution was determined. In Fig.5 shows the plot of the conditional mean  $\bar{h}_2 | h_1$  as a function of  $h_1$  is given together with the upper and lower standard deviation regions.





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For randomly distributed particles the moment  $E(h_1h_2)$  and the form of  $E(h_2)|h_1$  depend on the shape of particles. In the circular case it is  $h_1=h_2$  and  $E(h_2)|h_1$  is a straight line. The empirical relation in Fig. 5. shows deviations from the linear form, which indicate that some of the particles are slightly elongated as it can be seen in Fig.4. This particle shape characteristic is probably connected with interaction between the particles and matrix grain boundaries during annealing.

The mean  $\overline{h_1 h_2}$  calculated by a standard statistical procedure for twodimensional random variables (Sachs, 1978) is equal to 27.90 mm<sup>2</sup> being close to the value above which was obtained by the test quadrat K method. The corresponding covariance and the correlation coefficient are  $C_{12} = 5.52$ mm<sup>2</sup> and  $r_{12} = 0.80$ .

## CONCLUSIONS

A counting method for estimation of the moment  $E(h_1h_2)$  by equation (6) for two dimensional convex particles is proposed. The estimation procedure requieres counting the particles occupying the interior of the test quadrat K (the side length L of which should not be too large compared to the particle sizes) and estimation of the mean  $EN_K(A)$  as well as the estimation of the densities  $N_A$  and  $N_L$ . However, a sufficient number of measurements (different positions of the test quadrat) is necessary to get a reasonable accuracy.

The moment  $E(h_1h_2)$  can be taken as a measure of mean shape and size of two-dimensional convex particles.

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### REFERENCES

- Miles RE. The sampling, by quadrats, of planar aggregats. J. Microsc. 1978; 113: 257-267.
- Ryś J. Quantitative Beschreibung von heterogenen Karbidverteilungen in Stählen. Freiberger Forschungshefte. 1989; B 265: 142-154.
- Sachs L. "Statistische Auswerteverfahren". Springer, Berlin, 1978.
- Saltykov SA. "Stereometrische Metallographie". Deutscher Verlag für Grundstoffindustrie, Leipzig, 1974.
- Stoyan D, Kendall WS, and Mecke J. "Stochastic Geometry and Its Applications". Akademie-Verlag, Berlin and J. Wiley & Sons, Chichester, 1987.
- Wiencek K, Hougardy H. Description of the homogenity of particles arrangament. Acta Stereol. 1987; 6: 69-74.