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REVIEW PAPER

A SURVEY ON N_V STEREOLOGY FOR CONVEX PARTICLES IN METALLOGRAPHY

Jakub Bodziony[1], Kurt Wiencek[2], Jerzy Ryś[2]

[1] Strata Mechanics Research Institute of the Polish Academy of Sciences,
ul. Reymonta 27, 30-059 Kraków, Poland
[2] University of Mining and Metallurgy (AGH),
al. Mickiewicza 30, 30-059 Kraków, Poland

ABSTRACT

This paper surveys main results of N_V-stereology for systems of convex particles with comments to their practical applications in metallography.

Keywords: convex particle structure, random set, particle density, stereology, metallography.

INTRODUCTION

The subject of many microscopical studies are structures consisting of dispersed particles which are distributed uniformly in material space. The particle density N_V, which gives the average number of particles per unit volume, is a basic parameter of such structures. When N_V is known one can calculate, for example, average values of many global structural parameters per single particle, which may be of theoretical or practical significance.

In metallography N is usually measured by stereological methods, i.e., by indirect procedures based on measurements made on representative sections of three-dimensional structure being investigated. Sections are generated with known geometrical objects, so-called test objects, which are positioned at random in relation to the structure. Planes are the most popular test objects.

Probably, the beginning of N_V-stereology is connected with the appearance of some simple formulae which were developed for systems of spheres in the early 1930s by Tammann and Scheil (Saltykov, 1974). The following review is based on the properties of sections. Two types of sections will be considered:

(i) simple sections induced by a test object in the form of a finite system of separate points, a line, a plane or a three dimensional slice, and (ii.) parallel sections formed by a pair or a series of parallel simple sections.

Throughout the review it is assumed that the particles are convex. Consequently, N_V-stereology for simple sections is closely related to Cauchy's formulae, which are well known in integral geometry (Santaló, 1976). N_V-stereology for parallel sections, which was developed in the framework of the random set model, are presented only by showing its main results (Kraj, 1967; Wiencek, 1996).

The aim of this article is to present stereological equations in which N_V appears explicitly, so-called N_V formulae; it is not intended to give a complete review. Only such problems were taken into consideration

(i) which have a stable position both in stereology and quantitative metallography, i.e. which appear in such basic handbooks as in Underwood (1970), Saltykov (1974), Weibel (1980), Saxl (1989), Ohser and Lorz (1994), Ryś (1995), Kurzydłowski and Ralph (1995), and Stoyan, Kendall and Mecke (1995),

(ii) or which are recognized as original. (in the authors' opinion) or are results of recent studies, published in this issue.

BASIC FORMULAE FOR PARTICLE STRUCTURES

Geometrically, a particle, X, will be treated as a convex body. Its volume V, surface area S and total mean surface curvature M belong to the so-called convex body functionals (Bodziony, 1965; Santaló, 1976; Stoyan et al., 1995).

Let D be the mean projection length of X onto a random line and F the mean projection area of X onto a random plane. The Cauchy projection formulae (Santaló, 1976) are as follows:

$$S = 4F \tag{1}$$

and

$$M = 2\pi D \tag{2}$$

The particle structure Ξ is composed of non overlapping particles X_i (i=1,2, ...), which are distributed randomly in space R^3, forming a random set, a so-called germ-grain model (Stoyan et al., 1995; Ohser and Lorz, 1994).

For dispersions analysed in metallography, it is typical that $V_V < 10\%$ and $N_V > 10^6 \text{mm}^{-3}$, which means that the ratio of particle size (or size of the particle's nearest neighbourhood) to sample size (~10 mm) is near zero. Consequently, an unbounded and isometric (homogeneous and isotropic) random set is a realistic model.

Besides N_V, the structure Ξ is characterized additionally by some global parameters, namely the densities of the particle functionals given above (i.e. the average sum of particle functional per unit volume). These densities include volume density V_V, surface area density S_V and total surface curvature density M_V. These functional densities and N_V determine the mean global parameters of single particles, i.e., mean volume ⟨V⟩, mean surface area ⟨S⟩ and mean surface curvature ⟨M⟩ which satisfy :

$$\langle V\rangle = N_V^{-1} V_V \quad , \tag{3}$$

$$\langle S\rangle = N_V^{-1} S_V \tag{4}$$

and

$$\langle M\rangle = N_V^{-1} M_V \quad . \tag{5}$$

Because of the additivity of particle functionals, analogues of the Cauchy formulae are true also for the particle system Ξ, for the pairs of the means (⟨S⟩, ⟨F⟩) and (⟨M⟩,⟨D⟩), respectively. Then the Eqs. (3) - (5) may by rewritten

$$V_V = \langle V\rangle N_V \quad , \tag{6}$$

$$S_V = 4\langle F\rangle N_V \tag{7}$$

and

$$M_V = 2\pi\langle D\rangle N_V \quad . \tag{8}$$

Because Eqs. (6) - (8) contain the parameter N_V, they can be considered to be the basis of N_V-stereology for simple sections.

STEREOLOGY FOR SIMPLE SECTIONS

The following simple test objects will be used: a system of separate points P, a line G, a plane E and a slice T(t) of thickness t. The integer 1 will be assigned to a point which is a non-empty intersection of X with P. The intersection $\Xi \cap P$ forms a system of points in the space $R^0 = P$ with point density N_P. The parameters V_V and N_P satisfy Glagolev's formula (Saltykov, 1974)

$$V_V = N_P \quad . \tag{9}$$

A non-empty intersection of X and G is a segment (chord). The intersection $\Xi \cap G$ forms a system of chords in the space $R^1 = G$ with chord density N_P. The parameters S_V and N_L satisfy Saltykov's formula (Saltykov, 1974)

$$S_V = 4N_L \quad . \tag{10}$$

Finally, a non-empty intersection of X and E is a planar convex figure. The intersection $\Xi \cap E$ forms a system of convex planar particles in the space $R^2 = E$

with particle density N_A. The parameters M_V and N_A satisfy Bodziony's formula (1965)

$$M_V = 2\pi N_A \quad . \tag{11}$$

By means of Eqs. (6), (7), and (8), as well as Eqs. (9), (10), and (11), one obtains the following formulae for the parameter N_V:

$$N_V = \langle V \rangle^{-1} N_P \quad , \tag{12}$$

$$N_V = \langle F \rangle^{-1} N_L \quad , \tag{13}$$

$$N_V = \langle D \rangle^{-1} N_A \quad . \tag{14}$$

By means of (12), (13), and (14), it is possible to obtain two further formulae. With respect to $\langle D \rangle$ and $\langle F \rangle$, the structure Ξ may be characterised by the factor κ_2,

$$\kappa_2 = \langle F \rangle \langle D \rangle^{-2} \quad . \tag{15}$$

In general, κ_2 depends on particle shape and size. For equal spheres it is $\kappa_2 = n/4$. For regular particles of equal size (cube, cylinder, elipsoid, ...) κ_2 belongs to the interval $[0.5 \leq \kappa_2 \leq \pi/2]$ (Underwood, 1970). The first formula for the parameter N_V results from Eqs. (13)-(15) (Underwood, 1970; Kurzydłowski and Ralph, 1995):

$$N_V = \kappa_2 \frac{N_A^2}{N_L} \quad . \tag{16}$$

Next, the structure Ξ may also be characterised with respect to $\langle D \rangle$ and $\langle V \rangle$ by a factor κ_3 given by

$$\kappa_3 = \langle V \rangle \langle D \rangle^{-3} \quad . \tag{17}$$

As with κ_2, κ_3 depends on particle shape and size. For equal spheres it is $\kappa_3 = \pi/6$ and for regular particles of equal size there is the inequality $0.16 \leq \kappa_3 \leq \pi/6$ (Underwood, 1970). The second formula for the parameter N_V results from Eqs. (9), (12), (14), and (17):

$$N_V^2 = \kappa_3 \frac{N_A^3}{N_P} \quad . \tag{18}$$

It could be concluded that any information about κ_2 and κ_3 in (16) and (18) (obtained by estimation, experience or mode 1) opens the way from sections to N_V.

A non-empty intersection of a particle X with a slice T(t) of thickness t is a convex body (a particle or a convex part of it). Consequently, the intersection

$\Xi \cap T(t)$ is a random set of convex bodies in the slice. The projection of $\Xi \cap T(t)$ onto a plane E which is parallel to T(t) is a two-dimensional random structure characterised by the projection density $N_A(t)$ (the average number of projected particles per unit area). The parameters N_V, $\langle D \rangle$ and $N_A(t)$ satisfy the formula of Cahn and Nutting (1959)

$$N_V = \frac{N_A(t)}{t + \langle D \rangle} \quad . \tag{19}$$

Formula (14) can be considered as a particular case of (19), obtained for $t \to 0$. Furthemore, inserting (14) into (19) yields the following formula:

$$N_V = t^{-1}\left[N_A(t) - N_A\right] \quad . \tag{20}$$

The presented Eqs. (12)-(14), (16), and (18)-(20) are formulae for the parameter N_V. Only in Eq.(20), N_V is the exclusive parameter of the Ξ structure and as such it may be useful for N_P N_V-estimation. In the other formulae further 3D parameters appear. In general, an estimation of the means $\langle D \rangle$, $\langle F \rangle$ and $\langle V \rangle$ (or κ_2 and κ_3) from measurements on sections is not possible. However, some results for non-spherical particles of regular shape (cube, cylinder, ellipsoid, ...) are given in Fullman (1953), DeHoff and Rhines (1961), Underwood (1970), and Ohser and Nippe (1997).

If Ξ is a system of spheres, it is possible to estimate the means $\langle D \rangle$, $\langle F \rangle$ and $\langle V \rangle$ from measurements made on linear or planar sections. However, in the case of the projection of $\Xi \cap T(t)$ onto E, only an approximate estimation of $\langle D \rangle$ is possible. In Czyrska-Filemonowicz et al. (1998) one can find an example for N_V-estimation based on equation (19) where $\langle D \rangle$ was estimated from measurements made on thin foil TEM images.

Systems of spheres. Let D and d denote the diameters of sphere and circle section, respectively. The well-known Bach formula (1967) for diameter moments says

$$\langle d^k \rangle = I_{k+1} \langle D^{k+1} \rangle \langle D \rangle^{-1} \qquad \text{for} \quad k = -1,0,1,2,..., \tag{21}$$

where $\langle d^k \rangle$ and $\langle D^{k+1} \rangle$ are diameter moments of order k and k+1 respectively and

$$I_s = \frac{\sqrt{\pi}}{2} \frac{\Gamma\left(\frac{s}{2} + \frac{1}{2}\right)}{\Gamma\left(\frac{s}{2} + 1\right)} \qquad \text{for} \quad s = 0,1,2,... \quad . \tag{22}$$

For $k = -1$, $I_0 = \pi/2$ results from (21) (Fullman, 1953)

$$\langle d^{-1}\rangle = \frac{\pi}{2}\langle D\rangle^{-1} \quad . \tag{23}$$

Inserting (23) into (14) yields the following formula (Saltykov, 1974)

$$N_V = \frac{2}{\pi} N_A \langle d^{-1}\rangle \quad . \tag{24}$$

Formula (24) enables unbiased N_V-estimation from profile diameter measurements made on planar sections, unfortunately, with infinite variance (Watson, 1971). This so-called Saltykov's method of inverse diameters is still popular in metallography (Saltykow, 1974; Ryś, 1995; Kurzydłowski and Ralph, 1995).

Let l be the chord length of $\Xi \cap G$ and f (l) the probability density of the chord length. For a system of spheres the following relation exists:

$$\langle D^2\rangle^{-1} = \frac{1}{2}\left[\frac{df(l)}{dl}\right]_{l=0} \quad . \tag{25}$$

For spheres, the mean projection area satisfies $\langle F\rangle = \pi/4\ \langle D^2\rangle$. Consequently, from (13) and (25) the following formula for N_V is obtained (Hilliard, 1968)

$$N_V = \frac{2}{\pi} N_L \left[\frac{df(l)}{dl}\right]_{l=0} \quad . \tag{26}$$

It should be noted that the instable estimation of f (l) makes formula (26) difficult to be used in practice.

Equations (24) and (26) are true for every isometric system of spheres. However, in practice often additional information about the particle structure, Ξ is given. For example, in metallography, the size distribution of particles or grains could be known as an empirical result of previous measurements or of theoretical considerations. In many practical situations, a sphere diameter distribution of log-normal or gamma type can be used as a reasonable general model (DeHoff, 1965; Saltykov, 1974; Moran, 1971). For dispersed carbides in steels it was shown (Ryś and Wiencek, 1980) that the particle diameters follow the Weibull distribution with probability density (Gajek and Kałuszka, 1994).

$$f(D) = n\,a\,D^{n-1}\,exp(-aD^n) \qquad for \quad n \geq 1\,, a > 0 \quad . \tag{27}$$

In such a case N_V-estimation can be based on formulae (16) and (18) for which κ_2 and κ_3 are given by

$$\kappa_2 = \frac{\pi}{2} \frac{n\Gamma\left(\frac{2}{n}\right)}{\Gamma^2\left(\frac{1}{n}\right)} \tag{28}$$

and

$$\kappa_3 = \frac{\pi}{2} \frac{n^2\Gamma\left(\frac{3}{n}\right)}{\Gamma^3\left(\frac{1}{n}\right)} , \tag{29}$$

respectively.

In Stoyan and Wiencek (1991) formula (18) is given for $\kappa_3 = 0.857$ (which was calculated from (29) for $n = 3$) in connection with the Stienen model, a model for the dispersion of Fe_3C particles in steel.

A special case of the Weibull distribution (27) is the Rayleigh distribution, with $n = 2$. Here, κ_2 and κ_3 are both equal to 1. Examples of N_V-estimation by means of formulae (16) and (18) for Fe_3C dispersions in steel which follow the Rayleigh distribution are given in Ryś and Wiencek (1980) and Wiencek and Ryś (1998). It should by noted that when the carbide volume fraction is known (as a result of chemical analysis or earlier measurements), N_V-estimation by (18) and (9) can be reduced to the estimation ofprofile density N_A, which can be carried out by simple counting measurement made on planar sections alone (Wiencek and Ryś, 1998).

Systems of convex polyhedrons. A system of space filling convex polyhedrons can be used as a model for the grain structure of polycrystalline metals. In metallography the model of equal Kelvin polyhedrons is often used (Underwood, 1970; Ryś, 1995). In this case N_V-estimation can be based on formula (18) with $N_P = V_V = 1$ and $\kappa_3 = 0.419$. However, the Poisson-Voronoi model may be more realistic (Stoyan et al. , 1995; Ohser and Lorz, 1994). Here $\kappa_3 = 0.323$.

It was shown empirically that when grains of polycrystalline Fe. (as well as some other metals) were approximated by spheres (because $V_V = 1$ the spheres must overlap), the sphere diameter distribution follows the Rayleigh one (Eq. (27) for $n = 2$) (Hu, 1974). In such a case N_V-estimation can be based on formula (18) with $N_P = V_V = 1$ and $\kappa_3 = 1$ (formula (29) with $n = 2$), which corresponds to a formula given by Tammann (Saltykow, 1974).

STEREOLOGY FOR PAIRS OF PARALLEL SECTIONS

In the following, pairs of parallel test objects will be taken into consideration : pairs of points $P(\lambda)$, pairs of lines $G(\lambda)$, and pairs of planes $E(\lambda)$, which are all separated by distance λ. A pair of parallel test objects corresponds to a pair of parallel sections through the particle structure Ξ.

An intersection of a particle X with a pair of parallel objects may form: a pair of points, a pair of chords or a pair of plane figures. For given λ, a pair of parallel sections is characterized by the density of pairs of intersections, while for variable λ, a pair of parallel sections which depends on λ, is characterized by the so-called intersection pair density function (IPDF). Particular IPDF's are: the point pair density function $N_P(\lambda)$, the chord pair density function $N_L(\lambda)$ and the profile pair density function $N_A(\lambda)$.

The aim of Ξ-stereology for pairs of parallel sections is to derive equations which are satisfied by the parameter N_V and the IPDF's. The main results are provided below (Wiencek, 1996).

For a system of spheres, the stereological equation which is satisfied by the parameter N_V and the function $N_P(\lambda)$ is of the form (Wiencek, 1989)

$$N_V = \frac{2}{\pi}\left[\frac{d^3 N_P(\lambda)}{d\lambda^3}\right]_{\lambda=0} \quad . \tag{30}$$

if the derivative exists. The very instable estimation of the third derivative makes formula (30) rather impractical.

For a complex number s, the Mellin transformation M(s) of $N_L(\lambda)$ is given by (Dziubiński and Świątkowski, 1980)

$$M(s) = \int_0^\infty \lambda^{s-1} N_L(\lambda)\, d\lambda \quad . \tag{31}$$

For a system of spheres the following equation holds for N_V (Duvalian, 1972; Wiencek, 1996):

$$N_V = \frac{4}{\pi} M(-2) \quad . \tag{32}$$

An example of N_V-estimation for a Fe_3C dispersion in steel which is based on formula (32), is given in Wiencek (1996). Finally, for a convex particle system, there holds the following equation for parameter N_V and function $N_A(\lambda)$ (Wiencek, 1998)

$$N_V = -\left[\frac{dN_A(\lambda)}{d\lambda}\right]_{\lambda=0} \quad . \tag{33}$$

A more detailed description of the N_V-stereology discussed here is given in this issue (Wiencek, 1998).

The disector. For convex particles, N_V-estimation by the disector (Sterio, 1984) is closely connected to that by pair of planes P(λ) given above. An approximation of the derivative in (33) by the differential quotient for $d\lambda \approx \Delta\lambda$ leads to a formula which is equivalent to (20) for $t = \Delta\lambda$ In such interpretation, formula (20) expresses the disector principle of counting for the convex case. An example for estimation of grain density N_V for polycrystalline iron using the disector is given by Kurzydłowski and Ciupiński (1996).

In principle, disector gives a rule for counting also of noncovex particles (Sterio, 1984). In a more detailed description, a non-convex particle is characterised by the so-called Euler number (EN) χ (Ohser and Lorz, 1994; Stoyan et al., 1995). Consequently, a structure Ξ is characterised by the density of the EN, χ_V. DeHoff (1987) and Gundersen et al. (1993) described methods for χ_V-estimation by the disector. Recently, Ohser and Nagel (1996) have developed a more general EN-stereology for parallel sections. From their theory the following equation for pairs of planar sections results

$$\chi_V = \frac{1}{\lambda}\left[\chi_A(\Xi \cap E) - \chi_A((\Xi \cap E) \cap (\Xi \cap E_\lambda)_{-\lambda})\right] \ , \qquad (34)$$

where : χ_A (.) is the EN-density of a section set (.) and B_Z is the set B translated by z. Formula (34) shows that χ_V-estimation may be reduced to point counting measurement made on sections and their appropriate set-transformation. An example of χ_V-estimation for pre-eutectoid ferrite particles in steel (Zurek, 1994; Sachova et al., 1996) is given in Ohser and Nagel (1996).

From the point of view of the present paper, it is important to notice that for convex particles the χ_V-formula (34) reduces to an N_V-formula. In this case, for a given small $\lambda > 0$ the EN-density $\chi_A((\Xi \cap E) \cap (\Xi \cap E_\lambda)_{-\lambda}$ is approximately equal to the IPDF $N_A(\lambda)$ and (34) coincides with (20) for $t = \lambda$, which presents the disector rule. As a result, for $\lambda \to 0$ formula (34) becomes (33).

STEREOLOGY FOR A SERIES OF PARALLEL PLANE SECTIONS

In principle, Ξ-structure stereology for a series of parallel plane sections is similar to that for a pair of parallel plane sections. In spite of this similarity, due to the more complex test object it is being analyzed here separately and later, although, chronologically, this kind of stereology appeared earlier than N_V-stereology for pairs. In addition, the mathematics of a series, which is based on stochastic processes, is quite different from that of a pair.

The original approach was introduced into stereology by Kraj (1967). His ideas were then further generalized and developed by Bodziony and coworkers (Bodziony and Kraj, 1968; Bodziony et al., 1972).

The idea of this approach is as follows. Let E be a fixed plane in $R^3 \supset \Xi$. The plane E_t is parallel to E at a distance t. The intersection $\Xi \cap E_t$ is a system of convex figures which form a twodimensional particle structure in E_t. Let $T_t \subset E_t$ be a rigid quadrat of area A. N(t) is the number of $\Xi \cap E_t$ -particles which belong to T_t

(a particle belongs to T_t if its reference point belongs to T_t). Taking t as a variable, the function N(t) may be treated as a realization of a stationary birth-and-death stochastic process of a Markov type (Kraj, 1967). The fundamental characteristics of the process, i.e., the asymptotic number distribution (with variance σ^2) and the correlation function r (t) are binomial and. exponential, respectively. As a consequence, the following formula for N_V is obtained :

$$N_V = -\sigma^2 \frac{\ln r(t)}{At} \quad . \tag{35}$$

This equation is the basis of the so-called coupled plane sections method for N_V-estimation (Bodziony et al., 1972). An example of its application is given in this issue (Ryś and Wiencek, 1998).

CONCLUSION

The review presented shows that a lot of work has been invested in N_V-stereology. It seems that in stereology there is no other parameter to which so much study has been devoted. It is well known that N_V is a quite difficult parameter since, by contrast to V_V, S_V, and M_V (in the general case and even for convex particles), no equation exists which relates this parameter N_V (as a fundamental characteristic of a particle system) to quantitative characteristics of simple sections. Obviously, it is closely connected with particle system geometry in a broad sense, i.e., with its topology. In this situation most of the existing results in N_V-stereology are developed for special models of which the sphere model is the most popular.

In the authors' opinion most activities in N_V-stereology have come from metallography. This is probably connected with the fact that in metallurgy simple isometric structures are typical, which can be easily described by simple models. In the area of dispersed phases, the spherical approximation is still widely accepted. However, it is obvious that when the particle shape differs significantly from a sphere, such an approximation does not ensure a sufficient precision of the estimation in all cases. An alternative is N_V-estimation by parallel planar sections. Here, the disector ensures an unbiased result, at least in principle. However, this method requires the production of very thin parallel sections, which is often in metallography very difficult. In this situation, metallographers are primarily interested in developing methods which make it possible to estimate N_V from sections which can be generated in an easier way.

By no means N_V-stereology should be considered to be a closed area of investigation.

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