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ESTIMATION OF THE PARTICLE DENSITY N_V BY COUPLED PLANE SECTIONS

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ABSTRACT

If a particle system in a material's microstructure is considered as a random set with a particle density N_V, the parallel plane sections may be described by a stationary stochastic process. A stereological equation derived for this model by Bodziony and Kraj which satisfies the process parameters and the particle density N_V is a basis for the N_V-estimation by the coupled plane sections (CPS). Studies of a Nimonic alloy matrix grain structure in a series of parallel sections indicate the compatibility of the model with the structure. The N_V-estimation result obtained is comparable with that given by the disector.

Keywords: convex particles, random set, birth-and-death process, ergodicity, grain structure, quantitative metallography, disector.

INTRODUCTION

In some material microstructures a phase can exist in the form of particles which are arranged statistically uniformly in space. The particle density N_V is the main stereological parameter of the microstructure. N_V-estimation using a stereological method belongs to the ones which are the most time consuming in metallography. Most of the existing estimation methods were developed for systems of spheres (Saltykov, 1973; Ryś, 1995). For disperse phases ($V_V \ll 1$), the sphere approximation is in principle acceptable. However, for micro-structures in which the particle-grains are in form of polyhedrons which fill the space totally ($V_V = 1$), the sphere approximation is not sufficient.

In the more general case of a convex particles system, N_V-estimation is possible by a series of plane sections, provided the section structures are not statistically independent; they are correlated. A fundamental stereological method for N_V-estimation is here the so-called method of coupled plane sections (CPS) which was developed by Bodziony and Kraj in the years 1968-1972 (Bodziony and Kraj, 1968; Bodziony et al., 1972). Another method which appeared about 10 years later is the well known disector method (Sterio, 1984) which seems to belong more to statistical methods because it is based on counting measurements which are made directly in 3D space.

The main aim of the present investigations is N_V-estimation by the CPS for microstructures in which the grains are filling the space totally.

THE GENERAL MODEL

It will be assumed that an unbounded structure **Y** is formed of convex particles which are homogeneous and isotropically distributed in the space $\mathbf{R}^3$. In particular **Y** is set of polyhedrons of plane faces which are filling the space. Here, the Voronoi model can be given as an example (Stoyan et al., 1995).

Let **t** be an axis in the space $\mathbf{R}^3$ in which a point "o" is marked. An arbitrary point of **t** is at a distance t from o, i.e., $t \in \mathbf{R}$. A plane $\mathbf{E}_t$ is perpendicular to **t** at the distance t from o. $\mathbf{T}_t$ is a quadrat of the area A in $\mathbf{E}_t$, for which a vertex B belongs to **t**, see Fig. 1. The intersection $\mathbf{Y} \cap \mathbf{E}_t$ of **Y** with $\mathbf{E}_t$ is a plane structure of convex profile-particles in $\mathbf{E}_t$. To each particle in $\mathbf{Y} \cap \mathbf{E}_t$ a reference point (e.g., the center of mass) is assigned. It will be assumed that a particle of $\mathbf{E}_t$ belongs to $\mathbf{Y}(\mathbf{T}_t)$ if its reference point belongs to $\mathbf{T}_t$. $\mathbf{Y}(\mathbf{T}_t)$ is a plane structure which belongs to $\mathbf{T}_t$ and n (n = 0,1,2, ..., M) is the number of its particles (i.e., the reference points).

A significant assumption is that the structure **Y** will be considered as a realization of a homogeneous and isotropic random closed set (Stoyan et al., 1995) which will be called the random structure and denoted by **Y**, as well. The particle density N_V is a quantitaive characteristic of the random structure **Y**. For the random structure **Y** and given t, the random set $\mathbf{Y}(\mathbf{T}_t)$ is characterised by a random variable N_t which takes integer values n with the probability p_{tn}. The parameters of the probability mass function (PMF) p_{tn} are: the mean m_t and the standard deviation σ_t. Then, for a given τ the pair of random variables N_t and $N_{t+\tau}$ are characterised by the covariance $C(N_t, N_{t+\tau})$. For $t = 0$, the PMF, its parameters and the covariance will be denoted by p_n, m, σ and C(t) (since $\tau = t$), respectively.

For t, as a variable, the random set $\mathbf{Y}(\mathbf{T}_t)$ which depends on t is characterised by the random variable N_t which depends on t and which forms a discrete-valued stationary stochastic process $\{N_t, t \in \mathbf{R}\}$ which is continuous in the parameter t (Fisz, 1980).

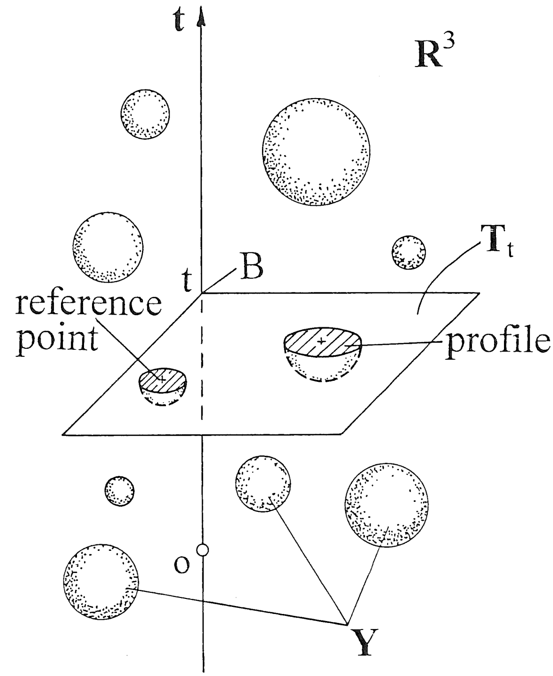

Fig. 1. Particles system **Y** and the test quadrat $\mathbf{T}_t$

A function N(t) which takes integer values n (n = 0,1, ..., M) and is continuous almost everywhere in $(-\infty, +\infty)$, is a realization of the process $\{N_t, t \in \mathbf{R}\}$. The stationarity is defined in relation to the functions: the first-order PMF p_{tn} and the correlation function $R(t,\tau) = C(N_t, N_{t+\tau})$ which do not depend on t, i.e., $p_{tn} = p_n$ and $R(t,\tau) = R(t) = C(t)$. This implies that $m_t = m$ and $\sigma_t = \sigma$. Some other properties of the function R(t) are as follows: for $\tau = 0$, $R(0) = \sigma^2$ and for $|t| \to \infty$, R(t) tends towards zero. The last one results from an intuitive physical interpretation, that if section structures are to be sufficiently far apart they are statistically independent. In practice, often a normalised correlation function, $r(t) = R(t)/\sigma^2$ will be used, where for t = 0, r(0) = 1.

The properties of a stationary process do not depend on the position of the origin o. Because the $\{N_t, t \in \mathbf{R}\}$ process is symmetric relative to o, its analysis can be limited to the part which belongs to the half space $\mathbf{R}_+ = [0, \infty)$, i.e., for $t \geq 0$. Therefore, it will be assumed that the random structure **Y** generates a stationary process $\{N_t, t \geq 0\}$ as a set of random variables N_t (numbered by a positive parameter t) of the same PMF's, p_n and of a correlation function r(t) with its properties given above.

Ergodicity

The function N(t) is a realisation of the stationary process $\{N_t, t \geq 0\}$. A stationary process is called ergodic if it is possible to express its characteristics by means of a single realisation, given in a form of a function N(t). In principle, ergodicity is a form of the law of large numbers for stationary processes which is of importance for statistical estimation procedures. An ergodicity, which is suitable for stereology will be expressed below.

Let for a given function N(t), a set $\mathbf{t}_n$ (n = 0,1, ..., M) in $\mathbf{t}$ be defined as follows $\mathbf{t}_n = \{t \in \mathbf{t}: N(t) = n\}$. Then, $l_n(t)$ is the length of $\mathbf{t}_n$ which belongs to the interval [0, t] of a length l. It will be assumed that a stationary process $\{N_t, t \geq 0\}$ is ergodic, when the PMF p_n can be expressed by (Bronstein and Siemiendiajev, 1959; Papoulis, 1965)

$$p_n = \lim_{l \to \infty} l^{-1} \int_0^l l_n(t)\, dt \tag{1}$$

and, for a given $\tau = \Delta t$ ($\tau \geq 0$), the correlation function $R(\tau)$ can be expressed by

$$R(\tau) = \lim_{l \to \infty} l^{-1} \int_0^l [N(t) - m]\,[N(t+\tau) - m]\, dt\,. \tag{2}$$

Equation (1) express the ergodicity in the PMF p_n while equation (2), in the correlation function R(t).

From a stereological point of view, a random structure **Y** will be a useful model for an empirical particles system when it generates a stationary ergodic process $\{N_t, t \geq 0\}$ which is ergodic in the PMF p_n and in the correlation function R(t), as well.

THE BIRTH-AND-DEATH PROCESS

Bodziony and Kraj (1968) pointed out that the general process postulated above can by modelled by a birth-and-death process which belongs to the class of discrete-valued stationary Markov processes for a continuous t parameter (Feller, 1961).

For a given t and the integers k, n (k, n = 0,1, ..., M), the so-called transition probability $P_{kn}(t)$ is the probability that $N_t = n$ with the condition that $N_{t=0} = k$. For a given k, there exists such a small distance t (denoted by Δt) that the random variable N_t almost always takes one of the three values: k + 1 (birth), k − 1 (death) or k only. A birth-and-death process is defined when the transition probabilities $P_{kn}(\Delta t)$ are given. A general formula for $P_{kn}(\Delta t)$ is as follows

$$P_{kn}(\Delta t) = \begin{cases} \nu(k)\Delta t + o(\Delta t) & \text{for} \quad n = k+1 \\ \mu(k)\Delta t + o(\Delta t) & \text{for} \quad n = k-1 \\ o(\Delta t) & \text{for} \quad n \neq k-1, k, k+1 \end{cases} \tag{3}$$

where $\mu(k)$ and $\nu(k)$ are functions of k and $o(\Delta t)$ tends towards zero faster than Δt. For the transition probabilities, given by (3), the limit $\lim_{t\to\infty} P_{kn}(t)$, if it exists, determines the so-called asymptotic distribution of the process. It can be shown (Gichman and Skorochod, 1968) that for the birth-and-death process above, the asymptotic distribution is equal to the stationary distribution p_n, which means, $\lim_{t\to\infty} P_{kn}(t) = p_n$. Consequently, for $t \to \infty$, $R(t) \to 0$ which is in accordance with the general model above.

Bodziony et al. (1972) have shown, that for the model above, a formula for the particle density N_V may be written

$$N_V = \frac{1}{2A} \sum_{n=0}^{\infty} \left[\nu(n) + \mu(n)\right] p_n, \tag{4}$$

where A is the area of $\mathbf{T}_t$. This means, the parameter N_V is determined by the model functions $\nu(k)$, $\mu(k)$ and p_n.

For a simple but important special case, the functions $\mu(k)$ and $\nu(k)$ are linear relative to k,

$$\begin{aligned} \nu(k) &= \nu - \lambda k \\ \mu(k) &= \mu k, \end{aligned} \tag{5}$$

where ν, λ and μ are positive constants. In this case the asymptotic distribution p_n is a binomial one, expressed by the formula

$$p_n = \binom{M}{n} p^n (1-p)^{M-n}, \tag{6}$$

where the parameters p and M are as follows

$$\begin{aligned} p &= \frac{\lambda}{\lambda + \mu}, \\ M &= \frac{\nu}{\lambda}. \end{aligned} \tag{7}$$

This means, the process $\{N_t, t \geq 0\}$ of the transition probabilities determined by (5) is a birth-and-death process with an asymptotic binomial distribution (Feller, 1961; Bodziony and Kraj, 1968). The correlation function r(t) is as follows

$$r(t) = \exp[-(\lambda + \mu)t]. \quad (8)$$

Finally, the formula (4) taking into consideration (5) and (6) results in a simple form (Bodziony et al. 1972)

$$N_V = A^{-1}(\lambda + \mu)\sigma^2. \quad (9)$$

It is important to notice that for large M, according to the de Moivre-Laplace theorem, the PMF p_n can be approximated by a Gaussian distribution. In such a case, the stationary Gaussian-Markov process $\{N_t, t \geq 0\}$ is ergodic relative to the condition given by the equations (1) and (2) (Bendat and Piersol, 1971; Stark and Woods, 1986). As a result, the formula (9) for N_V is a stereological relation in which the values λ, μ and σ, because of (1) and (2) can be considered as characteristics of parallel sections made for a given structure **Y**.

Some empirical remarks

It can be shown that some empirical results support the model above. In the metallography of materials with homogeneous particle systems usually the microstructure is analysed on one representative section. For a set of randomly positioned test quadrats **T** in the section plane, an empirical PMF for the number of particle intersections in **T** needs to be estimated. Assuming, that the structure follows the model above, because of stationarity and ergodicity, the empirical PMF can by considered as an estimate of the model PMF p_n.

An analysis of many data which are given for particles or grain systems in alloys shows that the empirical PMF p_n may by approximated by a binomial distribution. As an example, Fig. 2 and Fig. 3 show the empirical p_n function for the number of Fe_3C-particles (Wiencek and Hougardy, 1987) and ferrite grains in steel (Kasprzyk, 1990), in a test quadrat **T**, respectively, in comparison with the binomial PMF (6) in a Gaussian approximation.

In order to support this statement, Table 1. gives some additional information according to the empirical binomial parameter p (formula (6)), for Fe_3C-dispersions in a carbon steel (Fe – 0.6 % C, V_V = 9.5 %) of different dispersion degree and test quadrat size A. The dispersions were formed during a coarsening process at 700°C different times (with the passage of time the degree of dispersion became lower). All the analysed empirical PMF's p_n are of the binomial-type. (As an example in the Table 1., for the carbide dispersion which coarsened for 300 h, the parameter p of a binomial distribution, estimated by a test quadrat of area A = 20 mm × 20 mm is equal to 0.31.)

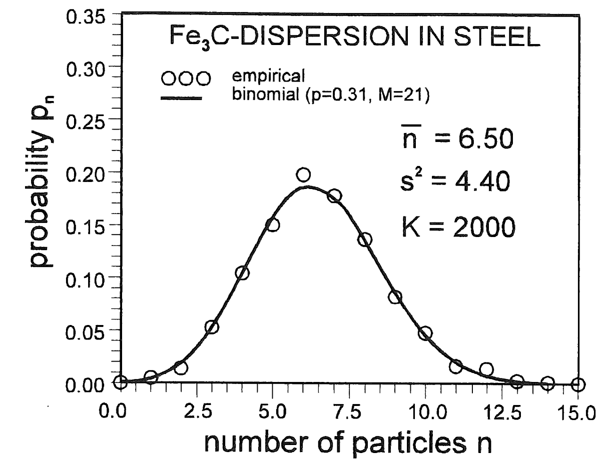

Fig. 2. Distribution of the Fe_3C- particle sections number in test quadrat.

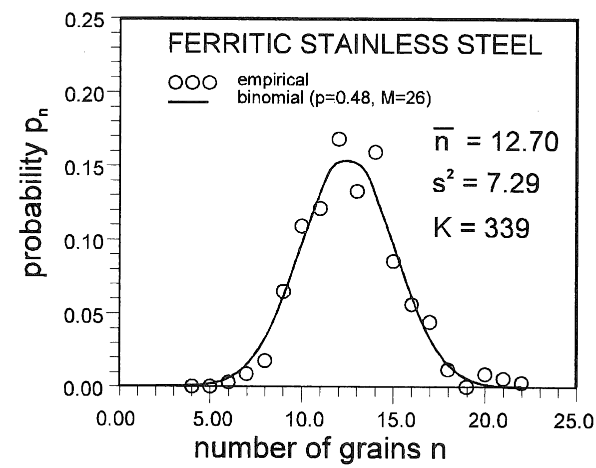

Fig. 3. Distribution of the ferrite - grain sections number in test quadrat.

Table 1. The p-binomial parameter of the distribution of the number of Fe_3C-particle sections in test quadrat.

test area A mm × mm	time h 100	time h 300	time h 600	time h 1200
10 × 10	0.27	0.30		
20 × 20		0.31	0.37	0.27
30 × 30				0.30
40 × 40				0.26

According to the data above, it may be supposed that the binomial distribution p_n is a typical one for material phases which appear in the form of particles or grains. A more detailed study (Stoyan and Wiencek, 1991) shows that the Fe_3C- particles are randomly distributed in the steel space, however, the sizes of the neighbouring particles are not statistically independent. There are spatial correlations which can result from some non-stochastic conditions which are imposed on the particle system, such as: (i) random distribution in the space, (ii) finite volume fraction $V_V >> 0$ and (iii) the particles do not overlap.
Unfortunately, it was not possible to find any empirical data on the correlation function R(t). Therefore, based on the empirical PMF p_n only, it could be stated, that from a $\{N_t, t \geq 0\}$ process point of view, the spatial correlations discussed above reflect in equation (3) by the parameter λ in (5). Consequently, the empirical particle systems can by described by a birth-and-death process with an asymptotic binomial distribution. It is worth noticing that for $\lambda \to 0$ the particle systems tend towards a Boolean model (Stoyan et al., 1995) in which the particle sizes are not correlated and now the asymptotic distribution is a Poisson one (Bodziony et al., 1972).

ESTIMATION

The estimation of the particle density N_V which is based on formula (9) is related to the estimation of the PMF p_n and the correlation function r(t) for a single realisation of the process $\{N_t, t \geq 1\}$, given in the form of a function N(t), by using equations (1) and (2).
Let $t_i = (i-1)\Delta t$, $(i = 1, ..., K;\ \Delta t > 0)$ be a series of K points in $\mathbf{t}$. The empirical N(t) function, which is determined by counting measurements made on a series of parallel sections, positioned in t_i and separated by the distance Δt, will be denoted by N(i). For a given N(i) function, the estimator m_1 of the parameter m may by calculated by the formula

$$m_1 = K^{-1} \sum_{i=1}^{K} N(i)\,. \tag{10}$$

Further, the estimator R(j) of the correlation function $R(\tau)$ in (2) for $\tau_j = j\Delta t$, $(j = 0,1,2, ...)$ is given by the formula

$$R(j) = (K-j)^{-1} \sum_{i=1}^{K-1} [N(i) - m_1][N(i+j) - m_1]\,, \tag{11}$$

where $N(i+j)$ denotes the $N(t_i + \tau_j)$ function. For $j = 0$, $R(0) = s^2$ estimates the variance σ^2. The normalised function r(j) is, $r(j) = R(j)/s^2$. It should be noted that the estimator given by (10) is unbiased, when the one given by (11) is asymptotic unbiased (Gajek and Kałuszka, 1994).

Next, for an exponential correlation function r(t) (8), the estimator of the sum $\lambda+\mu$ is equal to the slope of the estimated ln r(j) function. Finally, the estimator for the particle density N_V is given by (9) in which $\lambda+\mu$ is replaced by its estimator.

APPLICATION

These investigations were carried out on a recrystallized, coarse grained Nimonic alloy. Fig. 4 shows a microstructure, etched using Marble's etchant (Ryś et al., 1973) in which the matrix grains have a form which is typical for a polycrystalline metal.
As a result of the qualitative metallography the grains have been considered to be approximately convex polyhedrons with plane faces.

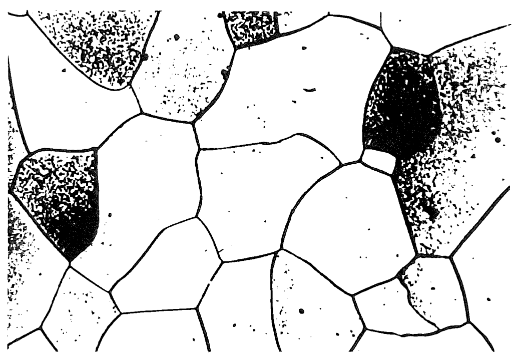

Fig. 4. Microstructure of the Nimonic alloy.

There are two reasons for the investigations performed: (i) comparison of the structure with the model, and then (ii) estimation of the grain density N_V by the CPS.

For the quantitative metallography four equal specimens (denoted I, II, III, IV) of a cuboidal form of 36.10 mm height and with a mean quadrate cross-section area of 8.09 mm^2 were prepared. A preliminary estimation of the grain size indicates that the nominal distance between neighbouring sections should be $\Delta t = 0.05$ mm; this is cca. 7 times smaller than the mean grain diameter. The coarse grain structure of the alloy used makes possible the measurement of Δt with an acceptable accuracy. For each specimen, 30 sections were made by mechanical grinding followed by precise polishing. The distance Δt between two neighbour sections was measured by an optical interferometer with a precision of 5×10^{-4} mm. The deviations of the parallelism were less than 5 %. Because of the difficulties with preparation of the sections, the measured distance Δt is not a constant value. Fig. 5 shows the distribution of the 116 measured distances Δt, which belongs to the interval [0.048, 0.063] with mean $\langle\Delta t\rangle = 0.054$ mm. The section microstructures were taken at $\times$ 30 magnification. The section microstructures which depend on the distance t_i were represented by four series of 30 sections or 120 sections totally. For a microstructure given in the i-th section, the number of grains N(i) was determined by the second variant of the Jeffries method taking into account Saltykov's edge correction.

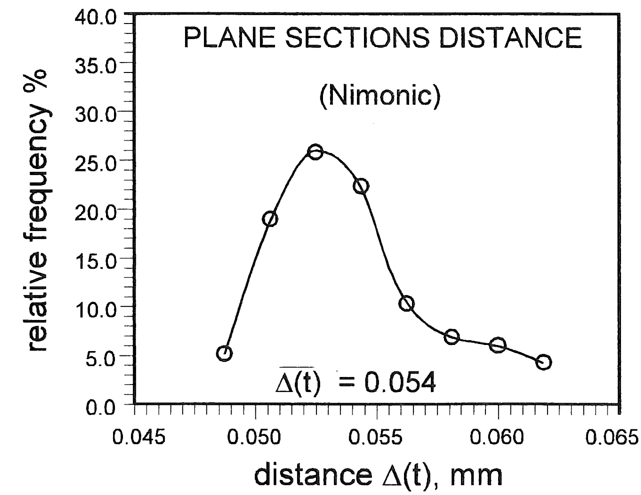

Fig. 5. Distribution of the Δt-distance between sections in the series.

Fig. 6 shows the empirical function N(i) (i = 1, ..., 120) (the particular series I to IV are marked) in comparison with the mean $m_1 = 43.2$ which was calculated by formula (10) for K=120. This empirical N(i) is to be considered as a realisation of a stationary stochastic process $\{N_t, t \in \mathbf{R}\}$.

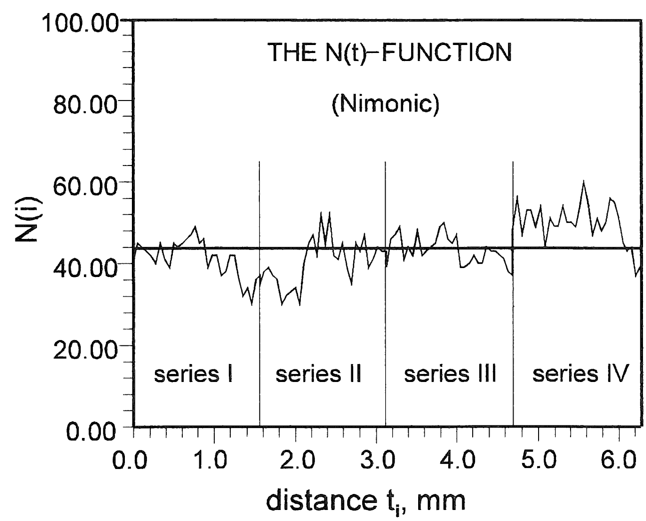

Fig. 6. The empirical N(t)-function for the Nimonic alloy.

The function p_n

The empirical p_n function was determined for the given function N(i) (i = 1, ..., 120), its statistical parameters are $m_1 = 43.20$ and $s = 5.90$. The appropriate binomial parameters p and M are $p = 0.20$ i $M = 223$, respectively. It should be noted that the p-value obtained is comparable with those of the carbide-dispersions in Table 1. Fig. 7 shows the empirical PMF p_n in comparison to the appropriate binomial one. Because of large M, the binomial PMF can be approximated by a Gaussian distribution, Fig. 7. A more precise statistical analysis with the χ^2-test indicates that the empirical PMF p_n can be considered as a binomial one.

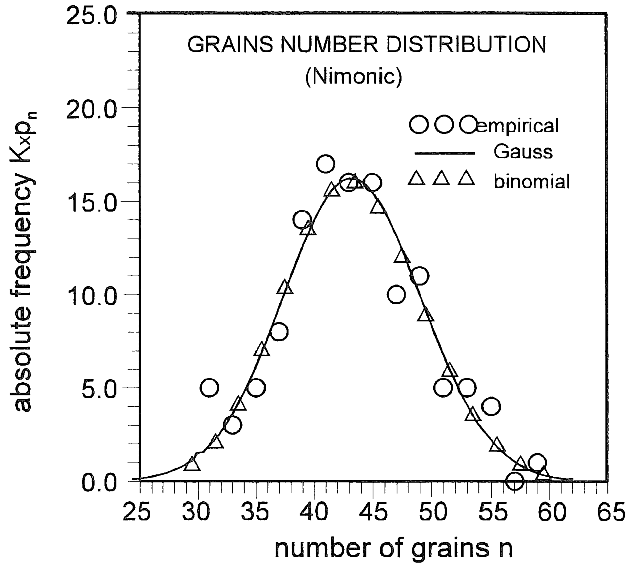

Fig. 7. Distribution of the number of Nimonic grains in the series of sections.

The correlation function r(t)

First, for the given N(i), (i = 1, ..., 120) the parameter m_1 was calculated by (10). Then, for $\tau_j = j\Delta t$, (j = 0,1, ..., 7) with $\Delta t = 0.054$ mm the empirical R(j) function was calculated by (11). Finally, by means of the empirical variance $s^2 = 34.81$, given for j = 0, the normalised correlation function r(j) was obtained. Fig. 8 shows the graph of ln r(j) and the approximation by a straight line. As a result, one can conclude that the empirical r(t) function is of exponential type.

The N_V-estimation

The estimator for $\lambda+\mu$ in (8) is equal to the direction coefficient of the regression line for the empirical ln r(j) function. Its value, determined by the least squares method is equal to

2.81 mm^{-1}. Substituting into equation (9), the estimators for $\lambda+\mu$ (2.81 mm^{-1}) and s^2 ($s^2 = 34.81\ mm^0$) as well as the mean area A of T_t (A = 8.09 mm^2) results in $N_V = 12\ mm^{-3}$.

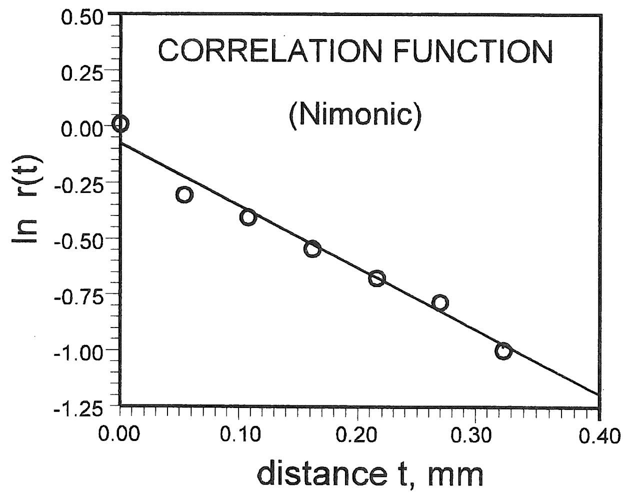

Fig. 8. The ln r(t) - plot for the Nimonic alloy.

In order to obtain information about the quality of the N_V-estimation given above, another estimation by a direct counting of grains in samples was performed. For the given function N(i), (i = 1, ..., 120) with the set of the Δt-distances between the subsequent neighbouring sections it is possible to determine the total number of grains in the sample investigated, by counting grains which appear or disappear on the subsequent sections. The appropriate formula, given by Bodziony (Ryś et al., 1972) is as follows

$$N_V = (2AL)^{-1}(n_a + n_d + 2n_{ad}), \qquad (12)$$

where: A and L are the section area and the height of the sample, respectively and then

n_a is the number of the grains which appear on the subsequent sections,

n_d is the number of grains which disappear from the subsequent sections,

n_{ad} is the number of grains which were intersected by the T_t only once.

For the sample investigated, the particle density N_V which was determined by means of formula (12) is, $N_V = 13\ mm^{-3}$. This value is comparable to that which is determined by the CPS. It should by noted, that the N_V-value obtained by formula (12) has a bias, caused by the fact that some of the grains with a size less than Δt could be not taken into account. However, for the coarse grained sample used, the fraction of such grains is not large.

It is worth also noticing, that the estimation method which is based on equation (12) is equivalent to the disector method (Sterio, 1984).

DISCUSSION AND CONCLUSIONS

The coupled plane sections method used is based on the following mathematical model. The convex particles structure is considered as a realisation of a random set which is characterised by the particle density N_V. The random structures which appear in parallel plane sections may by described quantitatively by a stationary ergodic Markov process, i.e., a birth-and-death process with an asymptotic binomial distribution and an exponential correlation function. Some process parameters are related to particle density N_V. Because of ergodicity, the N_V-estimation by the CPS method can be made as a single process realisation given in the form of a series of parallel sections. The results of the study show:

(i) The Nimonic alloy matrix microstructure follows the model. In particular, the empirical PMF is a binomial distribution (which is to be approximated by the Gaussian distribution) and the correlation function is exponential.

(ii) The result of the grain density N_V-estimation by the CPS is similar to that obtained by a disector-like method which gives also an idea on the quality of that estimation.

It should by noted that the accuracy of the CPS used for the N_V-estimation was not taken into consideration in this study. However, some remarks can be made. First, the precision of the estimation is related to the statistical nature of the estimators for the parameter m and the function R(t). Here, first of all, the sample size should be mentioned (the test quadrat size A and number of sections K). Next, the precision is influenced by: (i) the determination of neighbouring sections distance Δt, its realisation in practice and measurement, (ii) the Δt-distance distribution and (iii) the parallelism of the sections.

A detailed analysis of the precision by taking into account the factors above is a rather complicated problem. As an example, Fig. 5 shows the Δt-distribution in this study which is connected with the factor (ii). Nevertheless, because the difference between the estimation results obtained by the CPS and disector-like methods are not large, one can conclude that the factors above are of secondary importance in the present study. It seems, the precision of the CPS method could be effectively investigated by simulation.

Finally, some simple and useful conclusions can be drawn:

- The N_V-estimation by CPS is time-consuming but the result can be considered as reliable.
- The CPS method can be of importance in quantitative metallography when an approximation of the particle shape by a sphere is not acceptable.
- The particle (grain) size should be not too small.

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