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GRAPHITE PARTICLE SIZE DISTRIBUTION IN NODULAR CAST IRON

1. INTRODUCTION

The graphite particle size distribution is the fundamental statistic characteristics of nodular cast iron microstructure. The spatial microstructure is not accessible for direct observations, so its analysis is based on planar sections. Quantitative predictions according to particle size distribution from sections are made by stereological methods [1–3].

The aim of the present paper is an application of the parametric stereological method – which is based on the Weibull distribution, for estimation of graphite particle size distribution in nodular cast iron.

2. STEREOLOGY

A sphere of diameter D is the geometrical model for a graphite particle. A system of non-overlapping random spheres with sphere density N_V and volume fraction V_V is a model for the graphite particles in the cast iron microstructure. The sphere diameter distribution will be characterized by probability density function (PDF) f_3 and the mean $\langle D\rangle$. Planar section of a sphere is a circle profile of diameter d. The planar section of the random spheres is a profile system with profile density N_A and area fraction $A_A = V_V$. The profile diameter distribution will be characterized by the PDF f_2, the mean $\langle d\rangle$ and the harmonic mean $\langle d^{-1}\rangle^{-1}$. The stereological f_3-function estimation method is based on the Wicksell equation [4]

$$f_2(d) = \langle D\rangle^{-1} d \int_d^{D_m} \frac{f_3(D)}{\sqrt{D^2 - d^2}} dD, \quad \text{for} \quad 0 \le d \le D_m \tag{1}$$

where D_m is the maximum of D and d.

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The following formula exists

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

This means, the mean $\langle D \rangle$ in Eq. (1) is determined by a sections parameter. It is possible to transform Eq. (1) into an Abel type integral equation, which has analytical solution, given as an inversion formula for the unknown f_3-function. In metallography, the application of the inversion formula gives unsatisfactory results, therefore there is a need for the use of numerical solution method of Eq. (1). The well known numerical method is the Saltykov's successive substraction algorithm [1–4]. Unfortunately, this algorithm is unstable, i.e., small errors in the empirical PDF f_2 yield considerate errors in the calculated f_3-function, [4, 6]. One of the more stable procedures is the so-called parametric method [4, 5].

In the parametric method, for estimation of an unknown PDF f_3, a model PDF f_3 is chosen from a class of distributions, which depend on some parameters. A model f_3-function should be simple and the number of parameters low. Typical parametric model functions are PDF's of distribution: logarithmiconormal [7], gamma [8], Weibull or a mixture of Gaussian and exponential PDF's [5]. In stereology, the parametric method is realised by fitting the model profile PDF f_2 to the empirical one.

3. METALLOGRAPHY

The subject of this work is estimation of the f_3-function for graphite particles in nodular cast iron, whose chemical composition is given in Table 1. Figure 1 presents the microstructure in a planar section of the sample. The microstructure consists of approximately spherical graphite particles, which are randomly distributed in pearlit-ferritic matrix, in which the pearlite dominates.

Table 1. Chemical composition of cast iron

Element	C	Si	Mn	Ni	Mg	P	S
Content, wt. %	3.25	2.52	0.24	0.85	0.10	0.05	0.04

The quantitative analysis of the microstructure was performed on micrographs at × 520 magnification in 23 disjoint 100 × 100 mm test squares T. For $N = 1001$ particle sections the diameter (d) was measured by the TGZ-3 (Opton) particle size analyser. The basic stereological parameters of particle sections are: the area fraction $A_A = 0.144$ and the profile density $N_A = 966.68$ mm^{-2}. The statistical parameters are: the mean $\langle d \rangle = 0.0125$ mm, the harmonic mean $\langle d^{-1} \rangle^{-1} = 0.0092$ mm. The parameters of the graphite particles which are estimated by means of section parameters are: the volume fraction $V_V = A_A$, the mean diameter $\langle D \rangle = 0.0144$ mm (estimated by Eq. (2)) and the particle density $N_V = 67\,153$ mm^{-3} (estimated by the formula $N_V = N_A / \langle D \rangle$, [1]).

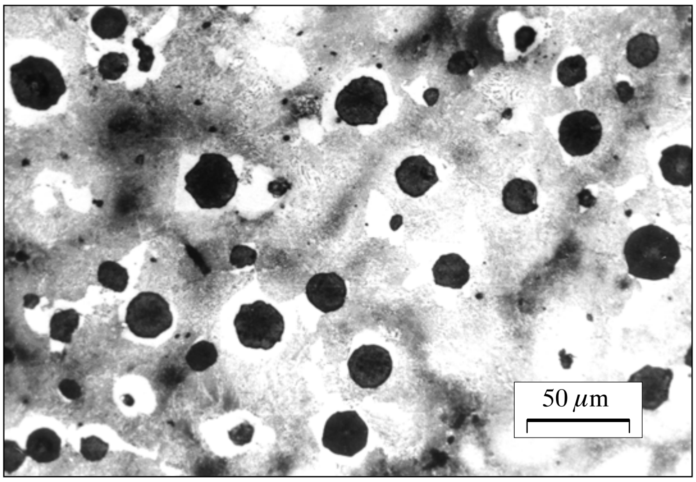

Fig. 1. *Microstructure of the nodular cast iron (etched by nital)*

4. SIZE DISTRIBUTION

According to the Saltykov algorithm, the [0, D_m] diameter interval is divided into k equal parts (classes) of Δ length. The discrete profile and sphere diameters are chosen as follows: $d_i = (i–1/2)\Delta$ and $D_j = j\Delta$, $(i, j = 1, ..., k)$. The respective PDF values are: $f_2(d_i)$ and $f_3(D_j)$. Figure 2 shows the empirical PDF's f_2 and f_3 – estimated by the Saltykov algorithm – for $k = 19$. A comparison f_3 to f_2 indicates a greater scatter of values while the f_3 is more irregular (especially for small D-values, e.g., the $f_3(D_1)$-value is negative). In such a situation a reasonable adjustment of the empirical f_3-function values aimed at prediction according to the graphite diameter distribution is, in principle, impossible. Therefore, for the graphite PDF f_3 estimation the parametric method was chosen.

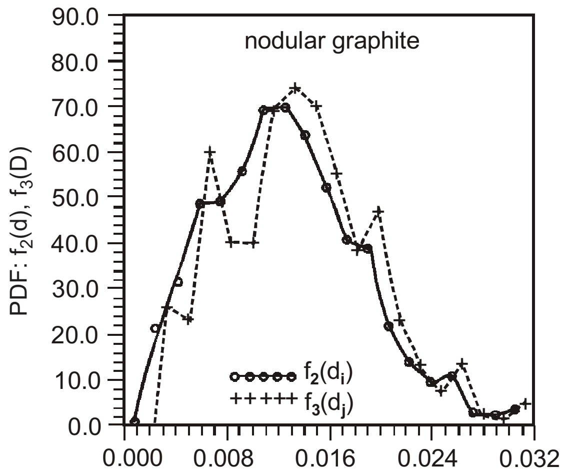

Fig. 2. *The empirical PDF f_2 and the PDF f_3 determined by Saltykov algorithm*

4.1. Parametric method

According to Anderssen and Jakeman “the use of parametric methods should only be made when independent arguments regarding their viability in a given context exist", [4]. Metallographical studies have shown, that in a ductile cast iron the graphite nodule diameter distribution follows the Rayleigh distribution which is a special case of the Weibull distribution [9]. The so-called Stienen model, i.e., a system of non-overlapping random spheres – in which the diameter distribution is Weibull distribution – was used as a model for carbide dispersions in steels [10, 11].

Due to the arguments above, the system of random spheres having Weibull distribution of the diameters seems to be a suitable model for the system of graphite particles in a nodule cast iron. Hence, for parametrization of the size distribution, Weibull distribution PDF may be chosen. The Weibull distribution PDF is of a form

$$f_3\langle D\rangle = n\alpha D^{n-1}\mathbf{exp}\left(-\alpha D^n\right) \tag{3}$$

where α and n are positive parameters, [12]. The α parameter may be expressed by $\langle D\rangle$ as follows

$$\alpha = \left(\frac{\Gamma\left(\frac{1}{n}\right)}{n\langle D\rangle}\right)^n \tag{4}$$

where Γ is the gamma function. Because of Eq. (4), n and $\langle D\rangle$ may be considered as parameters of the f_3-function given by Eq. (3). Substituting Eq. (3) in Eq. (1) gives the model profile PDF f_2. Estimation of a f_3-function by parametric method is reduced to the estimation of the n and $\langle D\rangle$ parameters of the model PDF given by Eq. (3). The estimation method is realised by fitting the model profile PDF f_2 to the empirical one according to a suitable criterion (maximum likelihood, least squares, etc.). For the parameters n and $\langle D\rangle$ the estimation by the least squares method (LSM) is used there.

4.2. Least squares estimation

Let y_i be the measured value of $f_2(d_i)$. The LSM function Q may be written in a form where n and $\langle D\rangle$ are the arguments and the values $f_2\,(n, \langle D\rangle; d_i)$ are calculated by Eq. (1) while taking into consideration Eq.(3) and (4). The minimum of Q, i.e., $Q_m(n, \langle D\rangle)$, determines the estimated parameters $(n, \langle D\rangle)$ for which the profile PDF f_2 gives the best fit to the data. The parameters estimated by LSM (which were determined numerically by computer for $k = 19$) are: $n = 2.75$ and $\langle D\rangle = 0.014$. Figure 3 presents the standard deviation function $s(n) = \sqrt{Q\left(n;\langle D\rangle\right)}$ for $\langle D\rangle = 0.014$. The parameters calculated by the model PDF f_3 are: the volume fraction $V_V = 0.147$ and particle density $N_V = 69049$ mm^{-3}. Table 2 presents the main parameters of the graphite phase which where estimated by means of section parameters (1) and by the obtained model PDF f_3 (2). Figure 4 presents the f_2-functions: the empirical and the model one (for $n = 2.75$ and $\langle D \rangle = 0.014$). It may be assumed, that the model f_2-function fits well the empirical one in the first approximation. Consequently, the graphite particles size distribution in the considered cast iron may be described by Weibull PDF f_3 with parameters $n = 2.75$ and $\langle D \rangle = 0.014$. Finally, Figure 5 shows the obtained model f_3 for graphite particles compared to the empirical f_3-function values estimated by the Saltykov algorithm. Although the scatter of the empirical values is significant it may be stated that the Weibull PDF f_3 adjusts the empirical data.

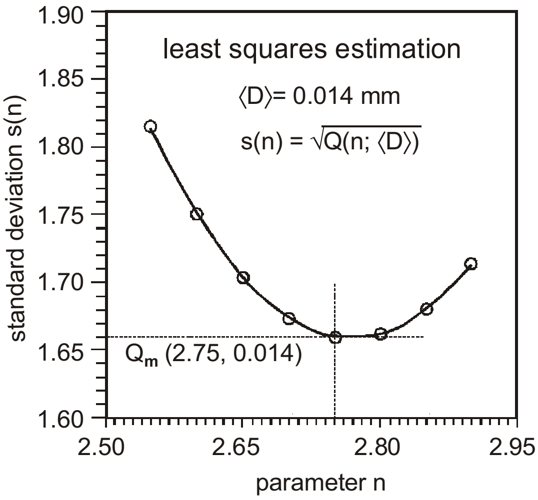

Fig. 3. *The least squares method* Q*-function*

Table 2. Parameters of the graphite phase which are estimated: (1) by section parameters, and (2) by the obtained model PDF f_3

Parameter / Method of estimation	$\langle D \rangle$, mm	V_V	N_V, mm^{-3}
(1)	0.0144	0.143	67192
(2)	0.0140	0.147	69049

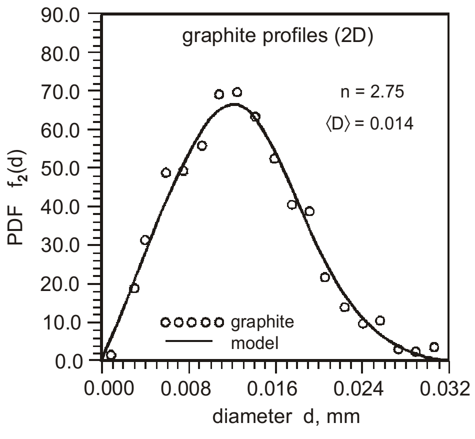

Fig. 4. *The empirical and model profile PDF f_2*

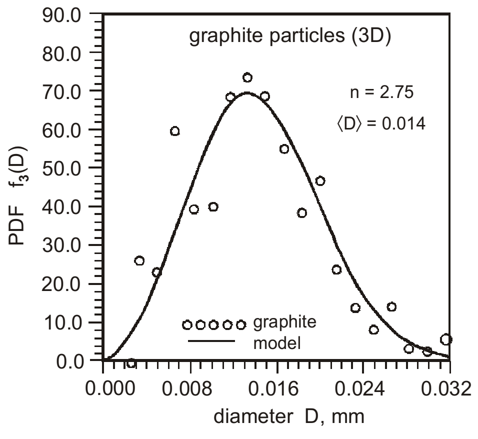

Fig. 5. *The model PDF f_3 and empirical PDF f_3 determined by Saltykov algorithm*

5. CONCLUSION

The PDF f_3 of graphite phase in the considered nodular cast iron may be described by the PDF of the Weibull distribution with the parameters n = 2.75 and $\langle D \rangle$ = 0.014. If a model PDF f_3 belongs to the PDF's class of Weibull distributions, its estimation by the parametric method may de based on fitting the model PDF f_2 for planar sections to the empirical PDF. In a special case when the n-parameter is known, the estimation of the mean $\langle D \rangle$ may be done by the following stereological equations

$$\langle D \rangle = \frac{2}{\pi n} \frac{\Gamma^2\left(\frac{1}{n}\right)}{\Gamma\left(\frac{1}{n}\right)} \langle d \rangle \tag{5}$$

and

$$\langle d \rangle = \frac{N_L}{N_A} \tag{6}$$

where N_L is the particle linear sections density.

From Eqs. (5) and (6) it results, that the estimation of the PDF f_3 of Weibull distribution with the known parameter n, made by the parametric method, may be reduced to an estimation of stereological parameters N_L and N_A, which is based on simple counting measurements of particle sections with suitable test objects, in particular, with test line (for estimation of N_L) and with test plane (for estimation of N_A).

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