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ANALYSIS OF GRAPHITE PARTICLE IN DIFFERENT NODULAR CAST IRONS

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ABSTRACT

In the study microstructure of nodular cast iron was analysed with a computer supported image analyzer. Research was done into the possibilities of describing the spatial nodule size distribution by a modified Saltykov method. The Saltykov method of describing spheres in space was modified by introducing a simple volume correction factor taking into account the real shape of the analysed nodules. The results are presented in tabular and graphical form and show that the modified Saltykov method can be successfully used for the determination of the spatial graphite nodule size distribution.

Key words: nodular cast iron, volume correction factor, graphite nodule distribution.

INTRODUCTION

It is known that the size and distribution of graphite nodules in the nodular cast iron matrix have a considerable effect on the efficiency of laser heat treatment by surface layer melting. In nodular cast iron, the graphite nodule cross-sections are easily seen on the polished surface of the prepared metallographical specimen. Computers are utilised in a variety of microstructural characterization functions - some are quite common, others are not. Treatments of stereological parameters measured on particles in the interior of a metalographic specimen my be found in a number of published papers (Rinaldi and Rossi, 1987; Shehata and Boyd, 1982; Vander Voort, 1991). Usually grain-size distributions were determined by measuring the area of individual grains in the plane of polish of metallographic specimens. Area measurement was used because the area of a non-equiaxed grain is independent of measurement orientation. Various types of grain-size distributions were constructed based on the nominal grain-diameter. Due to the fact that analysed particles frequently differ from global (ball or spherical particle) shape, the modified Saltykov method was used for analysis of nodular graphite in nodular cast iron (Vander Voort, 1984). For gathering the data on the microstructure, a CCD scanner camera was used supported by a software program for image analysis (Grum and Šturm, 1995). Measurements were made at a magnification of 100x on a microscope visual field surface of 0.2269 mm^2. CCD camera has high grade 512x492 pixels sensor, image scanner for overall focused images. On each specimen presenting different nodular iron quality, 30 visual fields were analysed, and the graphite particles with a section area greater than 30 μm^2 were measured. This minimum area of measured object was chosen on the basis of one pixel area (9 μm^2). Smaller particles do not contain enough pixels, therefore their measurement is not precise. On the other side, particles with cross-section area smaller than 30 μm^2, influence on the entire measured graphite area only with one percent of volume fraction. Figure 1 presents a typical microstructure of nodular cast iron with graphite nodules.

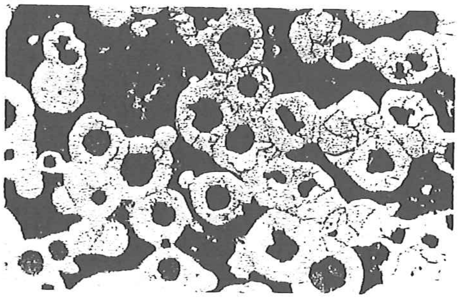


Figure 1. Microstructure of nodular cast iron 400-12, magnification 100x.

MATERIALS AND METHOD

The chemical composition of the investigated nodular cast iron qualities and some basic microstructure data obtained from computer analysis can be seen in Table 1. We usually define graphite particle size distribution in a unit of volume by three basic parameters, i.e.:

1. Number of particles per unit volume of alloy, N_V.
2. Mean particle diameter, $\overline{D}$.
3. Standard deviation of particle diameter, $\sigma(D)$.

Table 1. Chemical composition and microstructural components of nodular cast irons.

Iron composition	Nodular cast iron		
	400 - 12	500 - 7	600 - 3
C (%)	3.64	3.77	3.35
Si (%)	2.37	2.26	2.20
Graphite area fraction A_A (%)	12.0	10.8	9.6
Matrix structure	ferrite + pearlite	ferrite + pearlite	pearlite + ferrite

However, when we want to define the volume of grains or particles it is necessary to use an appropriate mathematical method which is based on statistical analysis of a sufficient number of analysed plane cuttings of a given specimen. The spatial graphite particle distribution can be calculated by mathematical models from the distribution of section areas of nodules assuming their shape (in our case spherical).

Mathematical methods for the calculation of the volume of spheres are based on the probability of sectioning plane cuttings through the plane between the exact centre and a sphere boundary to produce a range of planar sphere sizes. For the description of the spatial distribution of graphite nodules that have a typical spherical shape Saltykov method can be successfully used, (Vander Voort, 1984). According to this method, in determining the size of sphere diameters in particular classes a factor of 10^n was used. On the basis of measurements of graphite nodule diameters and identified maximum and minimum diameter we determined the exponent n as ranging within 0.8 and 2.1 in increments of $\Delta = 0.1$. Thus in our case 13 diameter size classes were used, i.e. from graphite nodule diameter of 6.3 μm to 125.9 μm. Section areas of graphite nodules were measured on a sectioning plane by an image analysis software program and they are classified into size classes according to their section diameters. The probability that a given nodule area falls within a given size class, at the known largest nodule diameter, was calculated according to the equation:

$$p(d_{i-1}, d_i) = \left[1 - \left(\frac{d_{i-1}}{d_{max}}\right)^2\right]^{\frac{1}{2}} - \left[1 - \left(\frac{d_i}{d_{max}}\right)^2\right]^{\frac{1}{2}} \qquad (1)$$

where: d_{max} - particle diameter of the largest class,
d_i and d_{i-1}- upper and lower diameter size in a particular class.

A general equation for 15 diameter size classes was developed for calculating the number of grains in a particular grain size class per unit volume $N_{V,i}$:

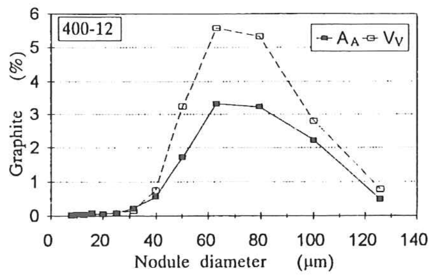
$$\begin{aligned} N_{V,i} = \; & (1.645 \cdot N_{A,i} - 0.4542 \cdot N_{A,i-1} - 0.1173 \cdot N_{A,i-2} - 0.0423 \cdot N_{A,i-3} - 0.01561 \cdot N_{A,i-4} - \\ & - 0.0083 \cdot N_{A,i-5} - 0.0036 \cdot N_{A,i-6} - 0.0019 \cdot N_{A,i-7} - 0.0009 \cdot N_{A,i-8} - \\ & - 0.00044 \cdot N_{A,i-9} - 0.00036 \cdot N_{A,i-10} - 0.0001 \cdot N_{A,i-11} - 0.00003 \cdot N_{A,i-12} - \\ & - 0.00003 \cdot N_{A,i-13} - 0.00001 \cdot N_{A,i-14}) / d_i \end{aligned} \qquad (2)$$

Where: $N_{A,i}$ - number of particles of i-th class per unit area,
d_i - maximum diameter for the particular class.
The coefficients in equation (2) were calculated as each grain contribution to each size class on the basis of the sectioning probability according to equation (1).

RESULTS

The results of the measured graphite area fraction A_A and the calculated graphite volume fraction V_V are shown with respect to volume portion of graphite in alloy in Fig. 2 as a frequency and a cumulative function of graphite nodule size class for nodular cast iron 400-12. Since in our case the shape of graphite nodules is not strictly spherical, as is assumed in the mathematical model, it comes to big deviations between A_A and V_V.

In order to achieve better calculation results of the volume fraction of graphite nodules V_V, Saltykov method was modified by introducing a correction factor for the shape of graphite nodules. Table 2 presents for each nodule size class in the nodular cast iron 400-12 the number of measured nodule sections and the calculated number of nodule sections per unit area $N_{A,i}$, and according to equation (2) the calculated number of nodules per unit volume $N_{V,i}$. On the basis of these information we can calculate the volume fraction of the graphite nodules in the alloy $\Sigma(N_{V,i} \cdot V_i)$, where $V_i = V_{i,max}$ is the maximal sphere volume in the i-th class. Table 2 presents also the corrected volume fraction $\Sigma(N_{V,i} \cdot V_i \cdot KF_{V,i})$ considering the nodule deviations from the shape of the sphere by introducing the volume correction factor $KF_{V,i}$.



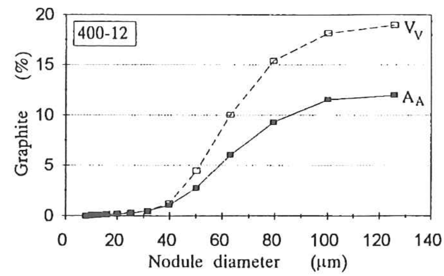


Figure 2. Distribution of the measured graphite area fraction A_A and the calculated graphite volume fraction V_V with respect to the nodule size class.

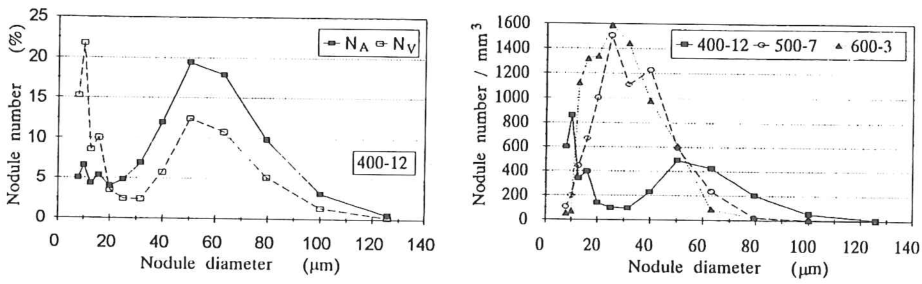
Table 2. Analysis of nodule sizes in nodular cast iron 400-12 considering the correction factor for the nodule shape.

Size class	Diameter (μm)	No. of observed nodule sections	$N_{A,i}$ (mm^{-2})	$N_{V,i}$ (mm^{-3})	$N_{V,i} \cdot V_i$ (%)	$KF_{V,i}$	$N_{V,i} \cdot V_i \cdot KF_{V,i}$ (%)
1	125.9 - 100.0	3	0.6	7.4	0.777	0.234	0.182
2	100.0 - 79.4	18	3.4	53.6	2.805	0.484	1.358
3	79.4 - 63.1	57	10.8	203.5	5.340	0.677	3.617
4	63.1 - 50.1	103	19.5	424.7	5.586	0.7	3.912
5	50.1 - 39.8	112	21.2	491.7	3.241	0.697	2.26
6	39.8 - 31.6	69	13.1	227.9	0.753	0.686	0.517
7	31.6 - 25.1	40	7.6	95.4	0.158	0.616	0.097
8	25.1 - 20.0	28	5.3	97.5	0.081	0.528	0.043
9	20.0 - 15.8	23	4.4	139.3	0.058	0.547	0.032
10	15.8 - 12.6	31	5.9	395.7	0.082	0.551	0.045
11	12.6 - 10.0	25	4.7	338.2	0.035	0.522	0.018
12	10.0 - 7.9	38	7.2	857.0	0.045	0.821	0.037
13	7.9 - 6.3	29	5.5	600.8	0.016	0.957	0.015
		Σ = 576	Σ = 109.2	Σ = 3932.7	Σ = 18.976	avg = 0.617	Σ = 12.133

$KF_{V,i}$ = volume correction factor.

The results of frequency distributions of $N_{A,i}$ and $N_{V,i}$ are shown in Fig. 3a with respect to the nodule size class for nodular iron 400-12. From the results in Fig. 3a we can conclude that there is a bimodal distribution of graphite nodules per unit volume. There are two size groups, i.e. nodules with average section diameters about 10 μm and 60 μm. Fig. 3b shows the distribution of nodule number $N_{V,i}$ per unit volume for different nodular cast iron qualities.

From Table 2 we can see that the calculated volume fraction $\Sigma(N_{V,i} \cdot V_i)$ is considerably greater than the measured percentage of graphite A_A = 12.0 % (Table 1). The deviations in graphite proportions obtained by the mathematical model occur due to irregular graphite particle shapes in space, i.e. due to form deviations of graphite nodules from the assumed spherical shape. We therefore proposed alterations in the calculation of the graphite volume according to the modified formula introducing the volume correction factor $KF_{V,i}$.



a) Frequency distributions of $N_{A,i}$ and $N_{V,i}$ b) Distribution of nodule number $N_{V,i}/mm^3$

Figure 3. $N_{V,i}$ analysis according to Saltykov method.

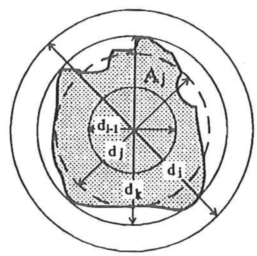
The volume correction factor gives ratio between volume of graphite nodule, which is expected on the basis of its section area, and volume of sphere circumscribed this nodule. From the measured graphite section area A_j we can calculate the diameter corresponding to an imaginary circle with equal area:

$$d_j = \sqrt{\frac{4 \cdot A_j}{\pi}} \tag{3}$$

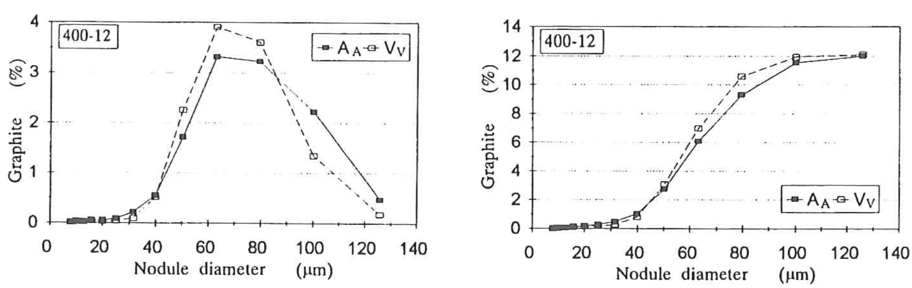
Then the volume correction factor for the determination of the real graphite particle volume can be written for each graphite particle in the i-th class in the following way:

$$KF_{V,i} = \frac{V_j}{V_k} = \frac{\frac{1}{6}\cdot\pi\cdot d_j^3}{\frac{1}{6}\cdot\pi\cdot d_k^3} = \frac{\left(\sqrt{\frac{4\cdot A_j}{\pi}}\right)^3}{d_k^3} = 8\cdot\left(\frac{A_j}{\pi\cdot d_k^2}\right)^{3/2} \qquad (4)$$

where: A_j - graphite section area in the i-th class,
d_j - diameter of the circle with an area A_j,
d_k - diameter of the circle circumscribed to the graphite area A_j,
V_j - graphite particle volume calculated from area A_j,
V_k - volume of the sphere which the analysed nodule with area A_j can entered into.



This means that each graphite particle section area and maximum diameter of this particle is measured. On the basis of these results we can calculate for each particle the correction factor and after this the entire graphite particle fraction in the precise size group. From the results of Table 2 we can notice that big graphite particles are of very irregular shapes. Smaller graphite particles become more spherical shaped. The value of the correction factor of graphite particles in the smallest group is influenced not only by real spherical shape of the particles, but also by resolution effect of microstructure magnification. Small graphite particles contain only some pixels and therefore seem to be spherical.



a) Frequency distribution b) Cumulative distribution

Figure 4. Distribution of the measured graphite area fraction A_A and the calculated graphite volume fraction V_V with respect to the nodule size class.

The final result of the calculation of the graphite volume fraction considering the volume correction factor for the graphite particle $\Sigma(N_{V,i}\cdot V_i\cdot KF_{V,i})$ = 12.1% deviates slightly from the measurements of the area fraction in the planar analysis. The results of the measured graphite area fraction A_A and the calculated graphite volume fraction V_V according to the modified Saltykov method are shown with respect to volume portion of graphite in alloy in Fig. 4 as a frequency and a cumulative function of graphite nodule size class for the nodular cast iron 400-12. Because of a big number of analysed graphite section areas in a section plane can be the measured distribution $A_{A,i}$ and calculated distribution $V_{V,i}$ very similar. Equality of cumulative results $A_A=V_V$ (DeHoff and Rhines, 1968; Kališnik, 1985) justifies the introduction of the volume correction factor into Saltykov method for determining volume distribution for particles, whose shape differs from perfect spherical shapes. Table 3 presents the final results of the analysis of graphite nodules for all the investigated nodular cast iron qualities according to the modified Saltykov method.

Table 3. Results of the analysis of graphite in different nodular cast iron qualities obtained by the modified Saltykov method.

Graphite nodule analysis	Nodular cast iron		
	400-12	500-7	600-3
Measured area fraction A_A (%)	12.0	10.8	9.6
Calculated volume fraction $\Sigma(N_{V,i} \cdot V_i \cdot KF_{V,i})$ (%)	12.1	11.1	10.0
Number of observed nodule sections	576	1161	1232
Mean graphite section area $\bar{A}$ (μm^2)	1099	536	444
Number of nodule sections per unit area N_A (mm^{-2})	109	201	217
Number if nodules per unit volume N_V (mm^{-3})	3933	7104	8633
Average nodule diameter $\bar{D}=N_A/N_V$ (μm)	27.7	28.3	25.1
Standard deviation of nodule diameter $\sigma(D)$ (μm)	24.6	12.6	12.0

From the results in Table 3 and from Fig. 3b we can note a similarity between nodular cast irons 500-7 and 600-3 in terms of the number and size of graphite nodules. On the other hand, nodular cast iron 400-12 differs considerably in having a much lower number of graphite nodules per unit volume N_V, which at the same area fraction means a higher number of graphite nodules of larger size. Much bigger standard deviations of graphite nodule diameter $\sigma(D)$ at almost the same calculated average diameter of graphite nodules $\bar{D}$ imply very non-uniform sizes of graphite nodules in nodular cast iron 400-12, which can be noted from graphite nodule distribution shown in Fig. 3b.

CONCLUSIONS

By the here proposed modification of the Saltykov method we can determine the volume distribution of graphite nodules which provides useful and reliable information on the microstructure of the investigated nodular irons. The advantage of the modified method is in a lower number of measurements required to measure graphite section area, still providing sufficiently reliable results on the volume distribution of nodules. At the beginning we have performed measurements on 30 measured fields. After that we have made measurements only on 3 measured fields, and the results differed only by 5%. The results of graphite nodule diameter distributions based on area analysis require a much higher number of measurements than the modified method, yet ensure equal reliability. It has been found or better proved that by introducing the volume correction factor we can accurately describe the proportion, size and distribution of nodular graphite per unit of volume especially when the nodules do not have a fully spherical shape. The results of the analysis on graphite nodules can serve as very useful information in predicting the microstructure conditions for laser melted and resolidified surface layer and is of significant importance in planning and optimizing the laser heat treatment process.

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