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Quantitative Analysis of Ductile Iron Microstructure – A Comparison of Selected Methods for Assessment

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Abstract

Stereological description of dispersed microstructure is not an easy task and remains the subject of continuous research. In its practical aspect, a correct stereological description of this type of structure is essential for the analysis of processes of coagulation and spheroidisation, or for studies of relationships between structure and properties. One of the most frequently used methods for an estimation of the density *Nv* and size distribution of particles is the Scheil - Schwartz – Saltykov method. In this article, the authors present selected methods for quantitative assessment of ductile iron microstructure, i.e. the Scheil - Schwartz – Saltykov method, which allows a quantitative description of three-dimensional sets of solids using measurements and counts performed on two-dimensional cross-sections of these sets (microsections) and quantitative description of three-dimensional sets of solids by X-ray computed microtomography, which is an interesting alternative for structural studies compared to traditional methods of microstructure imaging since, as a result, the analysis provides a three-dimensional imaging of microstructures examined.

Keywords: Ductile iron, Stereology, Scheil-Schwartz-Saltykov method, Microtomograph

1. Introduction

The key factors which set the criteria for the classification of engineering materials are quality indicators, taking into account directly or indirectly microstructural characteristics, expressed quantitatively by stereological parameters. These parameters describe, among others, the spatial structure of the set of solids. Quantitative assessment of the microstructure allows finding close relationships between structure and properties, and between the structure and the parameters of a technological process used for the alloy manufacture. The correct interpretation of a microscopic image, taking into account the shape and size of the individual elements of the microstructure, requires not only a precise definition of the morphological model, but often also a visualisation in the 3D scale. This issue is so important because a large share among the components of the microstructure of iron alloys have solids with a concave shape. Direct stereological relationships without proper diagnosis of the shape of a precipitate can be unreliable, even at the level of quality.

Procedures providing the information necessary to reconstruct the internal structure of opaque materials can be classified into two groups, i.e. destructive procedures, e.g. a sequence of metallographic specimens examined by optical microscopy [1, 2], the method of focused iron beam etching (FIB) - examinations using scanning electron microscope [3], and non-destructive procedures, e.g. X-ray computed microtomography [4-7].

In this article, the authors present selected methods for quantitative assessment of ductile iron microstructure, i.e. the Scheil - Schwartz – Saltykov method, which allows a quantitative description of three-dimensional sets of solids using

measurements and counts performed on two-dimensional cross-sections of these sets (microsections) and a quantitative description of three-dimensional sets of solids by X-ray computed microtomography, which is an interesting alternative for structural studies compared to traditional methods of microstructure imaging since, as a result, the analysis provides a three-dimensional imaging of microstructures examined.

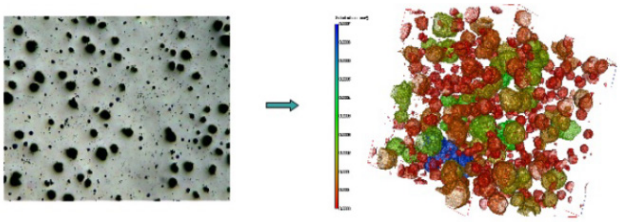


Fig. 1. (a) Microstructure with spheroidal graphite, metallographic specimen, (b) reconstruction of 3D graphite distribution in specimen of ductile iron

2. Test materials and methods

Low-alloyed nickel-copper ductile iron with about 1.9 wt% nickel content and about 0.9 wt% copper content was selected for tests. Chemical analysis of the melt was performed by the method of emission spectrometry using a POLYVAC 2000 apparatus. The results are summarised in Table 1.

Table 1.
The results of ductile iron chemical analysis, wt%

C	Si	Mn	P	S	Mg	Ni	Mo	Cu
3.60	2.45	0.32	0.035	0.020	0.065	1.90	-	0.93

The processes of heat treatment were performed in a Multitherm N41/M furnace supplied by NABERTHERM. In this furnace, the operations of austenitising were performed observing the following regime:

- heating with furnace to a temperature of 900°C,
- soaking at this temperature for 2 h.

The operation of isothermal cooling was carried out in a PEW-2 electric salt bath furnace (a mixture of potassium nitrate and sodium nitrite). The salt bath temperature and the time of isothermal cooling of cast iron amounted to 375°C / 2,5 h.

2.1. Measurement of dispersed microstructure by Scheil – Schwartz – Saltykov method

Stereological description of dispersed microstructure consists in estimation of the particle density N_V and size distribution (the size of the sphere is its diameter D) usually in the form of a probability density function $f(D)$. For the estimation of N_v it is necessary to make an assumption regarding the shape of particles. Most of the available methods are based on the assumption that particles are spherical in shape. The values of N_V and $f(D)$ are measured by the stereological methods based on direct measurement of the particle cross-sectional diameter (d), chord length (l) or area (a). These characteristics can be measured by image analysis methods. In practice, the description of dispersed microstructure uses one of the many approximate methods such as Scheil - Schwartz – Saltykov method, Spector method or Johnson method [8,9].

Scheil – Schwartz – Saltykov method (S-S-S)

The main problem in arranging the distribution of spherical particles according to their diameters is that spheres of different sizes give the circular cross-sections of the same size, but it is not always possible to assign the origin of these cross-sections to spheres of a specified size. Thus, a polydispersed particle system can be divided into several monodispersed systems (Fig. 2).

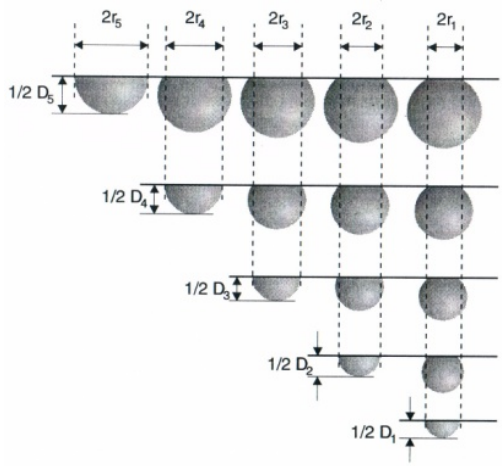


Fig. 2. Schematic diagram of a polydispersed system of spherical particles arranged into monodispersed systems

In the Scheil - Schwartz - Saltykov method, the elementary measurement is based on the particle planar section diameter (d). Based on the empirical distribution of planar section diameters $N_A(i)$, (i=1, 2, …, k) ($N_A(i)$ - the density of planar sections having a diameter in the range of $\Delta(i-1) \div \Delta(i)$, where Δ - the width of the class interval, i - the class number), a discrete distribution of the diameters of spheres in the $N_V(j)$ space is obtained (j=1, 2, …, k) ($N_V(j)$ - the density of spheres in space with a diameter Dj=Δj, i.e. the diameters of spheres D_i correspond to the right-side class boundaries in an empirical distribution of planar section diameters).

A relationship between the $N_A(i)$ and $N_V(j)$ distributions is represented by equation [9]

$$N_V(j) = \Delta^{-1} \sum_{i=j}^{k} \alpha_i N_A(i) \quad (1)$$

where: α – the array of coefficients.

A microsection of the ductile iron sample was prepared. Using a Reichert light microscope, the microstructure shown in the microsection was examined and photographed (Fig. 3).

On the images of cast iron microstructure (30 shots, the actual size of one image was 0.5 mm^2), the surface area of the planar

sections of the particles was measured using SigmaScanPro image analysis software.

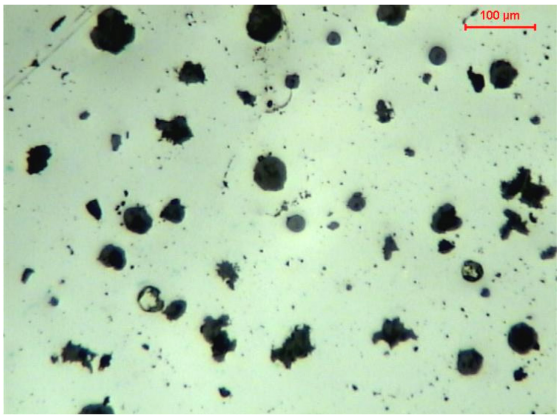


Fig. 3. Microstructure of ductile iron, unetched metallographic specimen

Then, equivalent diameters of the cross-sections of particles were counted (the diameter of a circle with an area equal to the area of the examined cross-section). Based on the equivalent diameter distribution of planar sections of the graphite particles determined by the Scheil - Schwartz - Saltykov method, the size distribution (D) of graphite particles was designed (Fig. 4).

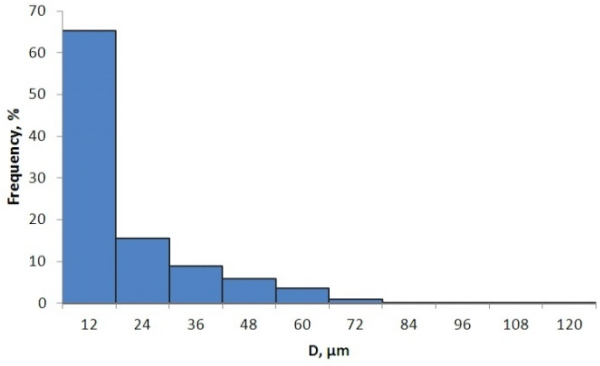


Fig. 4. Size distribution of graphite particles obtained by the Scheil – Schwartz – Saltykov method

2.2. Reconstruction of microstructure images in 3D scale using X-ray computed microtomography

X-ray computed microtomography (CT) is a non-destructive technique for visualisation of the internal structure of opaque solids, including information about their three-dimensional geometry and properties, using a set of two-dimensional projections, recorded at different angles relative to the coordinate system adopted. The mechanism of image formation in each layer, in the case of X-ray microtomography is based on the phenomenon of the radiation intensity weakening that takes place in each of the voxels, processed into contrast (grey scale) visible on the 2D images. Based on a direct relationship between the local grey level and the degree of radiation weakening, the mass distribution corresponding to the analysed volume is restored. Mathematical functional executing the task of the internal structure reconstruction of an object in 3D scale is based on Radon's theorem, which shows that the image of a 3D object can be reproduced from a finite number of its projections on the plane [10, 11].

Studies were carried out using a nanotom type apparatus for non-destructive testing by computed tomography. A series of images was taken during sample rotation by 360° in the field of view, at the angular increments of 0.25 ..... 0.5° at one stroke. These images contained information about the position and density of the object (voxel). The collected data were then used to reconstruct the numerical volume with the help of an algorithm that filters out the rear projection. The result was a 3D visualisation of the examined object (Fig. 5).

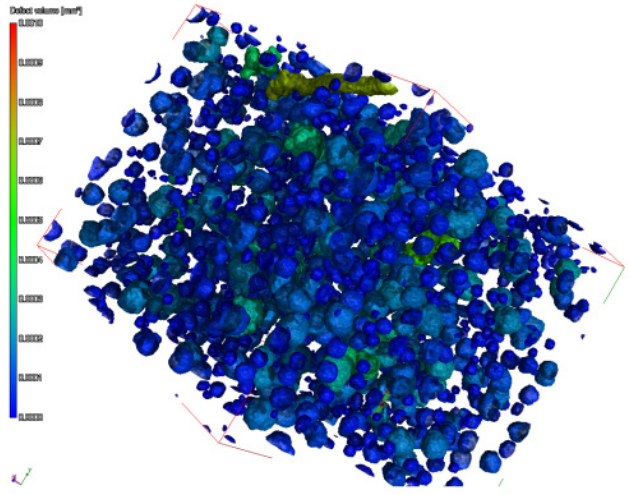


Fig. 5. The result of 3D imaging of ductile iron sample

The resolution of the method is mainly dependent on the assumed width of the scanned layers, which in turn (next to the size of the object and the physical properties of the material) affects the scan time. The resolution used in the present study was 1µm/voxel. The area reconstructed first and analysed next was ~ 1 mm^3. The data analysis was performed using a VGStdio MAX software. Figure 6 shows the size distribution of the graphite particles obtained by X-ray computed microtomography. In the case of the distribution obtained by X-ray microtomography, unlike in the case of the distribution obtained by the S-S-S method, no effect of increasing frequencies of lower classes due to the presence of concave particles was observed. This is due to the specific nature of 3D reconstruction - in the case of a concave particle, whose intersection with a plane can give more than one cross-section, a single particle is ultimately reconstructed.

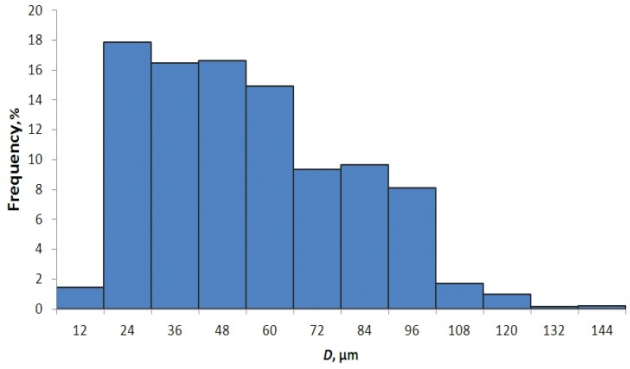


Fig. 6. Size distribution of graphite particles obtained by X-ray computed microtomography

3. Discussion of results

The diameter of the precipitates of the spheroidal graphite was estimated from a set of results of the measurements of the diameter of each particle in the field of view. The measurement results were obtained using different imaging techniques and various image analysis systems.

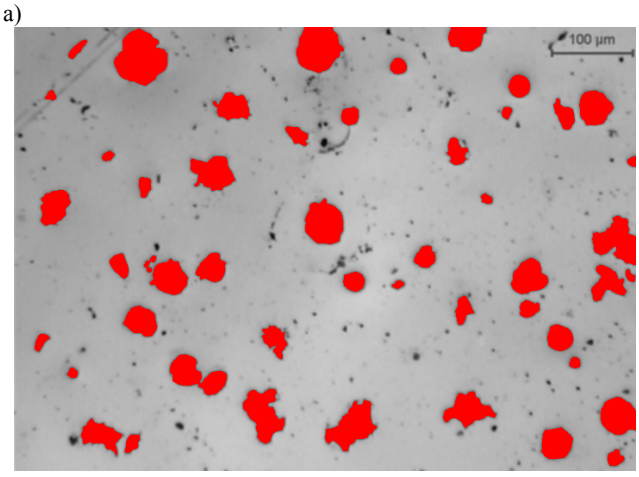
The distribution obtained from measurements using a light microscope has a very large first class, i.e. particles with a diameter of 0-12 μm represent about 65% of the cardinality of the entire set (Fig. 4), the fact that does not occur in the case of the results of imaging obtained by X-ray microtomography (here they make about 2% of the entire set- Fig. 6). The main reason for these differences is the fact that the registration of contrast for particles smaller than 10 μm was, in the case of microtomograph, beyond the resolving power of the detector. The imposed filters (median and erosion) also contributed to the rejection of particles with a minimum size treating them as noise or artifacts.

Another reason for the significant differences in the analysed distributions may be the shape of graphite particles, which in many places of the analysed microstructure takes the form intermediate between vermicular and spheroidal. The intersection with the microsection plane of a vermicular-shaped particle can give a number of sections of small diameter, treated by the image analysis software as separate objects, thus overstating the cardinality of the lower classes. In the case of the distribution obtained by X-ray tomography, no such effect is observed. This is due to the specific nature of 3D reconstruction - in the case of a concave particle, whose intersection with a plane can give more than one cross-section, a single particle is ultimately reconstructed.

The analysed distributions also differ in cardinality in classes of large diameters, but here a reverse situation occurs, and this time the results obtained by X-ray microtomography imaging show high cardinality in classes with large diameters. The assumed spherical character of particles, also used to reconstruct the size distribution of particles in the case of X-ray tomography, during analysis of the concave particles may be the cause of overstated cardinality of the upper classes. Since particle diameter (D) is calculated from its volume, in the case of the conglomerate particles (particles linked together - see Fig. 5), the result will be one diameter.

Considering the above, a comparison of the empirical distributions of the size of the graphite particles obtained by the analysed methods was presented, where only the particles with a diameter D> 10um were taken into account. Figure 7a shows an image of the microstructure of the examined cast iron with marked graphite particles selected for the measurement. The image shows a large number of fine particles that have not been classified for measurement. For comparison, Figure 7b shows the selected cast iron microstructure image projection made by X-ray microtomography. No particles of dimensions smaller than 10 μm are seen in this image which is due to a limited resolving power of the device.

a)



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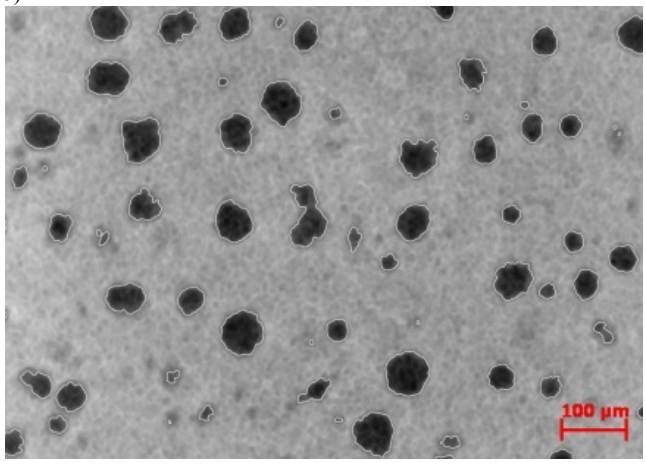

Fig. 7. An image of the microstructure of the analysed cast iron with marked graphite particles selected for the measurement (D>10 μm): a) light microscope b) X-ray microtomograph (tomogram)

Figure 8 shows a comparison of the empirical distributions of the size of the graphite particles obtained by the examined methods, assuming that only particles having a diameter D>10 μm will be analysed.

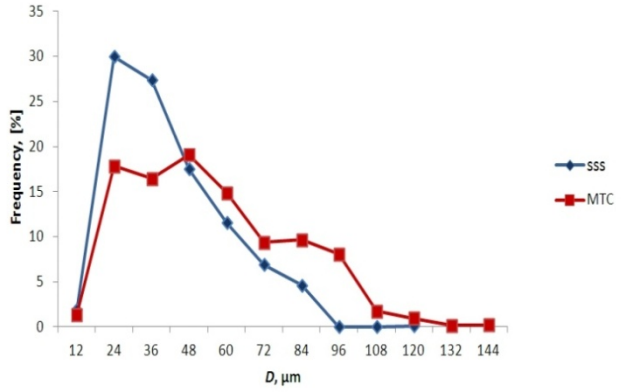

Fig. 8. Graphite particle size distributions obtained by the Scheil – Schwartz – Saltykov method and by the method of X-ray microtomography

The distributions shown above, though still characterised by some differences, are much closer to each other in shape. Significant differences are observed only in the extreme class intervals. In the case of particle diameter D>84 μm, the number of larger precipitates is definitely much higher in the microtomographic images, while in the case of the observations made by S-S-S, based on the microstructure analysis performed on a single metallographic specimen, the majority of the precipitates are particles with diameters smaller than D<36 μm.

4. Summary and conclusions

The study compares the selected methods for quantitative assessment of ductile iron microstructure. The measurement results were obtained using different imaging techniques and various image analysis systems.

Measurement of the size of selected particle (estimated by diameter or chord) to a large extent depends on the category of solids, to which this particle can be assigned (convex or concave). In the case of both methods analysed, a significant impact on the accuracy of the particle size distribution determination had, besides the size of particles, also their shape, which assumed an intermediate form between vermicular and spheroidal.

Because of the detector resolving power, the method of X-ray microtomography is applicable in the case of low dispersions of graphite particles in ductile iron or, due to the fact that it raises the probability of detecting particles of maximum size, it can be an excellent complementary tool to conventional imaging methods and analysis of microstructures.

An analysis of real images of three-dimensional structures is expected to allow an accurate assessment and defining a number of useful parameters (number of objects, topological characteristics, size of the object, the distance between objects, etc.) that can not be directly assessed by analysis of 2D images. Having available the images of the actual structure, it is also possible to simulate the load application and mechanical tests, and thus provide interesting information about the mechanical properties without the need to damage the sample.

The 3D imaging and analysis of 3D images is an innovative methodology that brings enormous benefits of both cognitive and exploratory nature.

Acknowledgements

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