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Short Report

On the Problem of Real and Apparent Size of Graphite Nodulus in a Ductile Iron

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Short Report

Nodular (spheroidal) cast iron, also called Ductile Iron (DI), is still of great importance due to the areas where it is used on a large scale, such as sewerage pipes, pipelines, etc. Among the Fe-C-Si system castings, DI is characterized by its higher ductility and its microstructure, in which free carbon is present in the form of nearly spheroidal grains. Depending on the variation in chemical composition and heat treatment, some carbon may appear in the combined form of cementite (Fe_3C) and some pearlite in the metal matrix. The formation of nodular graphite is the result of changes in the chemical composition of the alloy caused by ladle treatments called inoculation (addition of Fe-Si) and nodularization (addition of Mg). The role of Mg in DI is quite complex. Unlike Si, Mg is not a graphitizing element, it acts in the opposite direction, stabilizing cementite. However, when Mg is added to the metal bath and reaches its critical temperature, it evaporates, increasing the free volume of the metal bath and facilitating the formation of graphite. Mg also reacts with impurities such as S and O, thus initiating the process of desulphurization and deoxidation of the metal bath and forming thin layers of complex oxides and silicates, which act as a substrate for the heterogeneous nucleation of graphite, facilitating its subsequent growth by stacking basal planes in the c-direction, resulting in the formation of nodules [1-4].

Many studies show a strong correlation between the microstructure and the physical and mechanical properties of DI and highlight the significant role of the size of the nodules, their morphology and their distribution in the metal matrix. Quantitative analysis is therefore a fundamental tool for the quality control of DIs, since by quantifying the structural constituents, it is possible to predict the mechanical behavior of the material [1-5]. However, it is important to note that on the surfaces of the specimens prepared for metallographic analysis, the graphite cores and nodules are randomly cut and the dimensions observed in the image may not accurately reflect the actual size of the nodules. The scientific interest and practical importance motivated us to analyze the graphite nodules extracted from a DI by dissolving the metal matrix (iron) in order to assess their real size.

The material used for the study was a DI ingot obtained as a result of the chemical treatment of the Base Metal (BM) melted by inoculation with Fe-Si and nodularization with Mg and cast at 1362 ^oC in a sand ingot 10 minutes after treatment. The chemical composition of the MB and DI analyzed is given in Table 1. A 2 g sample of DI was dissolved in a solution of HCl + H_2O (80 ml + 80 ml) and heated between 50 and 90 ^oC, according to the method described in the paper [6]. The constituents of the residue were analyzed by SEM techniques using a SUPERSCAN/SS500-50 Shimadzu. The DI from the same bath was prepared for metallographic analysis, including grinding, polishing and chemical etching with 2% Nital. The DI samples were sectioned, metallographically prepared and analyzed by Optical Microscopy (OM) techniques using a Neophot-32 microscope.

Table 1: Chemical composition of liquid base metal and DI before casting.

Composition (% p)						
BM	**C**	**Si**	**Mn**	**P**	**S**	**Mg**
BM	3.86	2.18	0.21	0.08	0.015	---
DI	3.72	2.53	0.21	0.07	0.006	0.07

Images of the microstructure of DI analyzed by OM in its polished and chemically etched state are shown in Figure 1. One can see in Figure 1 the graphite nodules and cores surrounded by ferrite grains and pearlite grains in the regions furthest from the graphite. The quantitative assessment of the graphite nodules in this study was carried out using unetched micrographs at 100x magnification under bright-field microscopy and the graphical capabilities of Power Point software. The analysis was carried out by superimposing circular ovals of known diameter on the nodules, as shown in Figure 2. The size of the nodules was determined by overlapping known size circles-ovals on the calibrated micrographs. The results were statistically analyzed to determine the mean, standard deviation and absolute error of the measurements using a Student's Coefficient of 1.96. By processing the different OM images and using 60 measurements, the average size of the nodules was determined to be 37.54 ± 30.66 μm with a standard deviation of 15.64 μm. After dissolving the DI for 3 hours, washing and drying, the solid residue contained 3 components: graphite spheres (black, fine), a yellowish-grey mass in flakes and pieces of material that had not yet dissolved. The constituents were analyzed by SEM. In this work, the graphite nuclei were evaluated.

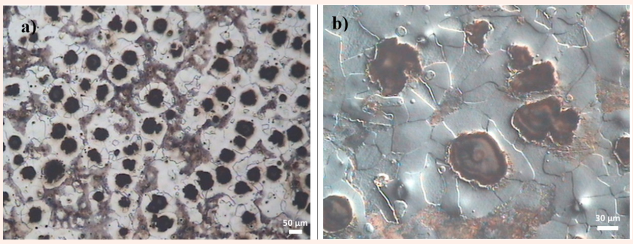

Figure 1: Microstructure of DI: a) 50x; b) 200x. OM.

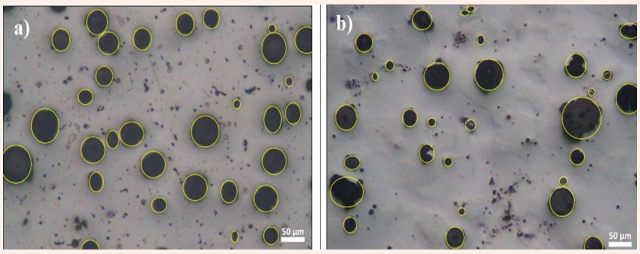

Figure 2: Characterization of graphite nodules in DI by OM, bright field: a, b) 100x.

Figure 3 shows SEM images of graphite nodules. It can be seen that the nodules are almost spherical in shape, fairly uniform, with some variation in size. Even finer particles of different shapes were seen. These are probably cementite flakes, part of the pearlite and graphite nuclei. Based on the 57 SEM measurements, the mean nodule size was determined to be 69.32 ± 19.38 µm, with a standard deviation of 9.89 µm. A significant difference between the average nodule sizes evaluated in the OM samples and in the SEM images can be explained by at least two reasons. First, the metallographic assessment evaluated the sizes of the nuclei, whereas the SEM images only evaluated the nuclei. And secondly, and most importantly, the SEM images assessed the diameters of the nodules and not their cross-sections.

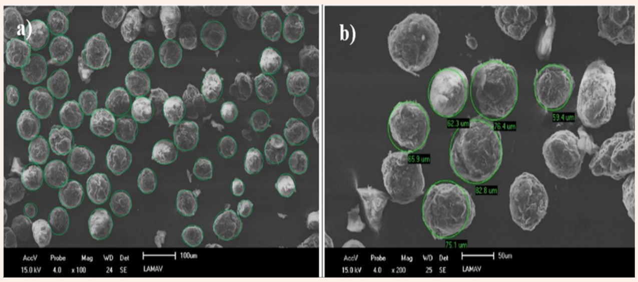

Figure 3: Images of graphite nodules extracted from DI through dissolution in HCl solution: a) 100x, b) 200x. SEM

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