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TEM OBSERVATION OF SPHEROIDAL GRAPHITE IN DUCTILE CAST IRON

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1. Abstract

This research produced typical ductile cast iron, and observed spheroidal graphite in this ductile cast iron by electron microscopes. For scanning electron micrographs of graphite morphology, the metallic matrix was dissolved away by deep etching in a 10% nitric acid solution. The result observed by scanning electron microscope (SEM), spheroidal graphite was patterned indented surface. High magnification image of the spheroidal graphite was observed graphite flakes sticking to surfaces. The cross-sectional transmission electron microscope (TEM) specimens of spheroidal graphite were prepared by Focused Ion Beam (FIB) system method. TEM dark field image using strong graphite (0002) diffraction was obtained from cross-section of the central part in spheroidal graphite, and it was found that a growth structure of a cone in spheroidal graphite radiated to outwards from the central part along the direction of c-axis of the graphite structure. In addition, with a cone radiated to outwards from the central part, graphite structure slightly rotated along c-axis of itself. Cross section of the outside part in spheroidal graphite was observed polycrystals whose c-axis of the graphite structure was the same direction.

2. Introduction

Over 60 years has passed since ductile cast iron was found [1]. From that time extensive studies to further improve ductile iron properties and to develop new casting practices and strategies have resulted in slimmer designs which had developed ductile iron, produced a variety of ductile cast iron with special mechanical, and other properties [2]. Now ductile cast iron is broadly used for auto parts, construction machinery parts and so on, because it has the characteristics, such as outstanding mechanical properties [3]. Result of many research studies, sulfur, oxygen contents and cooling rate were found important factors of the spheroidizing mechanism [4-6]. Researchers have proposed many theories about the spheroidizing mechanism: surface energy [7], nucleation [8], dislocation [9], bubbles [10] or adsorption [11]. Thus the spheroidizing mechanism has still not been totally clarified. And various models have also been suggested for growth of spheroidal graphite including interface breakdown model [11], circumferential growth model [12], growth by conical spirals [13], and Maltese cross pattern growth [14] yet there is no unanimity on growth mechanism of spherical graphite from the melt. In this work, as a part of the research which clarifies the nucleation and the growth process of spheroidal graphite, its graphite was investigated by electron microscopes.

3. Experimental

Casting was prepared in a 100 kg high frequency induction furnace. Melt was superheated to 1784 K. The magnesium treatment was performed using the"Sandwich Technique" for producing ductile cast iron. The ferrosilicon alloy containing 4.3% Mg and 1.6% RE was used in the spheroidising treatment. The casting were inoculated with 0.4 mass% of the charge with FeSi alloy (75% Si). Table 1 lists the actual chemical composition of casting in this study. The melt was poured into the 25 mm Y-block sand mold. In order to evaluate whether or not the casting was typical ductile cast iron, it was carried out microstructural examination and mechanical tastings. Characterizations of spheroidal graphite in the matrix were carried out on polished surfaces of samples using standard metallographic techniques and analysis software (Quick Grain, Inotech). The optical microscope was used BXSIM-DIC (Olympus). Samples were then etched with 1% nitric acid solution and microstructures of their matrices were characterized. For characterization of spheroidal graphite, five images were analyzed and the nodule count, nodularity and average sphere equivalent diameter of spheroid graphite as well as their size distribution were measured. Tension tests were carried out using a 30T Amsler type testing machine. Impact tests were done using of a 300J Charpy impact machine. Hardness tests done using of a Brinell hardness tester. For scanning electron micrographs of graphite structure, the metallic matrix was dissolved away by deep etching in a 10% nitric acid solution. The cross-sectional TEM specimens of the spheroidal graphite were prepared by FIB. The microstructures of spheroidal graphites were observed by a SEM with S-3500H (Hitachi, Co., Ltd.) and TEM with Topcon EM-002B.

4. Result and discussion

As the results of microstructure analysis, the number of spheroidal graphites in casting, nodule count, was 243 nodules per mm^2, average sphere equivalent diameter of spheroid graphite was 25.4 μm, and nodularity was 91.4 %. As the results of mechanical tastings, tensile strength was 471 MPa, elongation was 22.1 %, impact energy was 114 J, and Brinell hardness was 159 HB. Those results were normal values of ductile cast iron. Thus the casting produced this study was typical ductile cast iron. Figure 1 shows the SEM images of the sample. Figure 1 (a) shows microstructure of ductile cast iron after the metallic matrix was dissolved away by deep etching in a 10% nitric acid solution. Figure 1 (b) and (c) show low and high magnification image of the spheroidal graphite. The result observed by SEM, spheroidal graphite was not smoother but rougher surface. And, surfaces of spheroidal graphite observed sticking graphite flakes. Its size was about 2 μm. Figure 2 shows the TEM images observed for a cross-section of the central part in spheroidal graphite. Figure 2(a) and (b) show bright field image and dark field image using strong graphite (0002) diffraction. A cross-section of the central part in spheroidal graphite looked contrast difference (inside and outside of a red circle Fig.2(a)). Illuminated regions in this dark field image show fans, and they look like connecting at their apices. The direction of c-axis of the graphite structure in a cone was the same as radial direction. It found that growth structure of a cone in spheroidal graphite radiated to outwards from the central part along the direction of c-axis of the graphite structure. The results observed a cone region, with a cone radiated to outwards from the central part, graphite structure slightly rotated along c-axis of itself. Figure 3 (a) and (b) show the TEM images observed center of cross section of the central and outside parts in spheroidal graphite. Cross section of the central part in spheroidal graphite was mosaic. It was observed polycrystals whose c- axis of the graphite structure was the same direction

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5. Summary

・The growth structure of a cone in spheroidal graphite radiated to outwards from the central part along the direction of c-axis of the graphite structure regardless of whether nucleus-like core actually exist.

・With a cone radiated to outwards from the central part, graphite structure slightly rotated along c-axis of itself

.・Cross section of the outside part in spheroidal graphite was observed polycrystals whose c-axis of the graphite structure was the same direction.

6. References

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Table 1. Chemical composition of the alloy

(mass%)

C	Si	Mn	P	S	Cu	Mg	Fe
3.43	**2.37**	**0.40**	**0.02**	**0.01**	**0.17**	**0.03**	**Bal.**

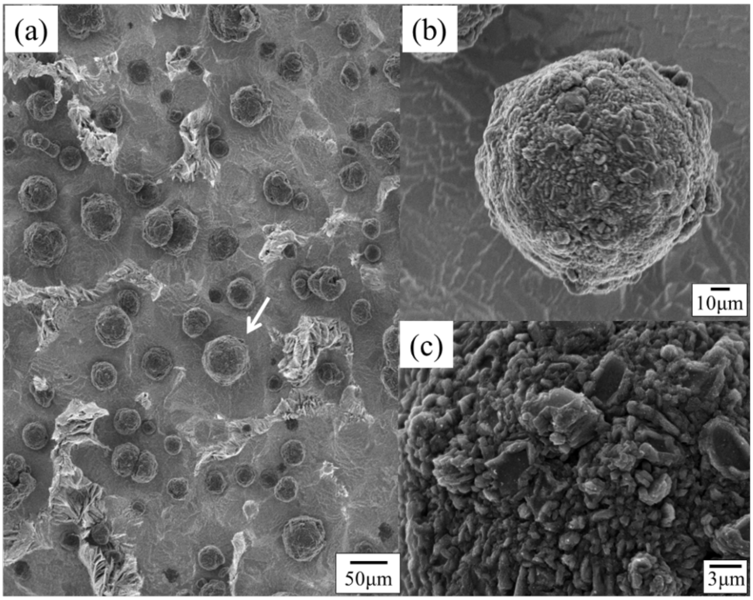

Figure 1. SEM images of ductile cast iron: (a) microstructure, (b) and (c) low and high magnification image of the spheroidal graphite.

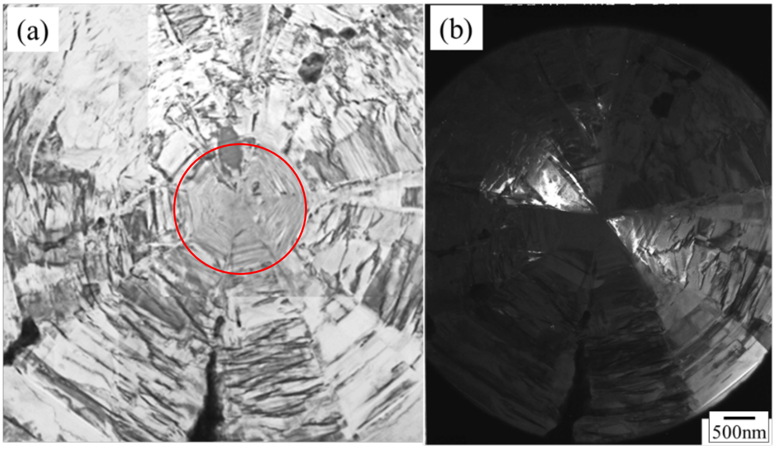

Figure 2. TEM images of cross-section of the central part in spheroidal graphite: (a) bright field image and (b) dark field image using strong graphite (0002) diffraction.

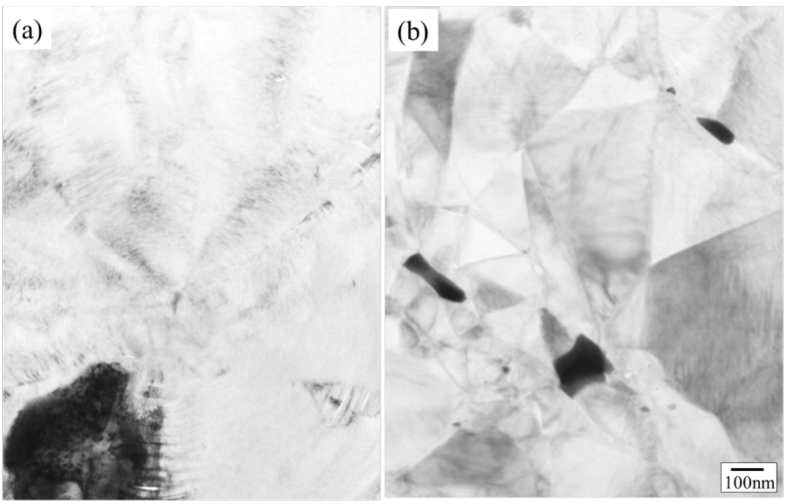

Figure 3. TEM bright field images: cross sections of the central (a) and outside (b) parts in spheroidal graphite.