Quantitative Characterization of Graphite Morphology in Cast Iron from 3D Perspective

Zhijun Ma\textsuperscript{1,a,*}, Zhong Yang\textsuperscript{1}, Yongchun Guo\textsuperscript{1}, Jianping Li\textsuperscript{1,b,*}, Minxian Liang\textsuperscript{1} and Shaogang Wang\textsuperscript{2}

\textsuperscript{1}School of Materials and Chemical Engineering, Xi’an Technological University, Xi’an, China.
\textsuperscript{2}Institute of Metal Research, Chinese Academy of Sciences, Shenyang, China

Email: \textsuperscript{a}9216053@qq.com; \textsuperscript{b}546223001@qq.com

Abstract. Compared with 2D microstructure characterization, quantitative characterization of graphite morphology from 3D perspective can make it more possible to predict and improve properties of cast iron with relatively high accuracy. In the present study, based on 2D metallographic photos, X-ray tomography was utilized to investigate 3D morphology of graphite clusters in different cast iron and 3D quantitative characterization of graphite morphology was carried out by Avizo software. The results show that basically all nodular graphite in ductile iron are isolated and almost all graphite phase is interconnected in gray iron. The interconnectivity of compacted graphite iron is closely related with vermicularity. The flake-shaped graphite in gray iron is different with coral-shaped graphite in compacted graphite iron from 3D perspective. The quantity proportion of graphite clusters smaller than 10000 \(\mu\text{m}^3\) is as high as 99.7\% and 97.1\% respectively in gray iron and compacted graphite iron with 91.3\% vermicularity. The volume of graphite clusters larger than 10000 \(\mu\text{m}^3\) basically accounts for most of graphite volume and the proportion is over 95\% in gray iron and compacted graphite iron with high vermicularity. The difference of graphite volume distribution among different cast iron is mainly concentrated in the volume range from 10000 to 100000 \(\mu\text{m}^3\).

1. Introduction

In spite of the birth, development and application of many advanced new materials in previous decades, the cast iron with over one hundred years history is still a preferred material used in cylinder head for high power density diesel engine [1-2]. For one thing cast iron is obviously better than aluminum alloy from the perspective of the volume size, corrosion resistance and strength, or the friction coefficient of the diesel engine, for another the higher castability is necessary because cylinder head is a typical thin-walled complex casting [3]. The unique mechanical and physical properties of cast iron have awarded the material such desirable and increasing demands in both automotive and locomotive industries [4-8].

It is generally recognized that the size and morphology of the graphite phase in cast iron play a crucial role in determining its various properties. Therefore, plenty of research has been done to examine and characterize the morphology and distribution of the graphite phase and some results are obtained [8-11]. In current research and industrial production to evaluate the properties of cast iron relies mainly on the subjective comparison of 2D metallographic images and ASTM/ISO Standard images. However, the answer to the nature of the 3D spatial structure of the graphite within cast iron isn’t given. The related research and development are not sufficient because its acquisition and quantitative characterization are difficult, for example traditional deep etching method [12] or the
serial sectioning method [13]. Some new progress has been made in recent years to address these issues [14-21], such as the 3D characterization of graphite morphology by destructive FIB-tomography [16-17] and X-ray tomography (XRT) [19-21].

Up to now the relationship between microstructures and properties of cast iron is sometimes controversial because the quantitative characterization of the graphite morphology and distribution with sufficient statistics is quite difficult. In the present study, XRT is utilized to investigate 3D morphology and distribution of graphite in gray iron, ductile iron and compacted graphite iron (CGI) with different vermicularity. The difference among them is discussed and quantitatively analyzed according to XRT statistics. These studies can make it possible to predict and improve properties of cast iron with relatively high accuracy.

2. Experimental Procedure

The morphology and distribution of the graphite phase can be manipulated through the combination of techniques such as changing the chemistry of the melt and altering the type and application of inoculant treatment. The samples used in the present study included gray iron, ductile iron and CGI. And there are five CGI samples with different vermicularity (52.8%, 66.5%, 76.1%, 83.9% and 91.3% respectively). The samples from J.P. Li group in Xi’an Technological University[22] were EDM-cut from the bottom half of the wedge block (as shown in Fig.1) into Φ1.0×5.0mm cylindrical rods.

![Figure 1. The schematic diagram of the wedge block of cast iron](image)

2D morphology can be observed by Leica DMI3000M optical microscopy (OM). Compared with 2D morphology, 3D morphology can characterize the microstructure of materials more comprehensively. The 3D morphology of graphite phase in cast iron was investigated by Xradia Versa XRM-500 XRT at Institute of Metal Research. It facilitates non-destructive observation of the internal structure of materials. The XRT experiment was carried out at 80kV accelerating voltage which ensured the measurement has sufficient intensity resolution to make a distinction between the iron and graphite phase. The resolution of present experimental setup is 0.7μm in the pure absorption contrast regime.

During the tomographic measurement, the sample was mounted at the rotational center of a high precision rotation stage and a series of 901 radiographic images were taken at 0.4° intervals during a 360° rotation of the sample. The entire diameter of the sample within the field-of-view of the detector during data collection, which took 12s for a given radiographic images. The 3D internal structure of the probed volume was reconstructed with the voxel resolution 1.2422×1.2422×1.2422μm³. A 450×450×800 voxels unit cell was extracted from every columnar sample to process and analyze 3D morphology of graphite phase quantitatively using Avizo software.
3. Results and Discussion

3.1 Morphology Characterization of Graphite in Cast Iron

Figure 2. 2D morphology of graphite in ductile iron observed by (a) OM and (b) XRT

Fig.2 shows the 2D image of ductile iron observed by OM and XRT respectively. The graphite 2D morphology obtained by two methods is basically the same. In order to reconstruct the graphite 3D morphology, graphite needs to be rendered. The boundary of the graphite is difficult to determine during rendering of the graphite because the resolution of XRT image is considerably lower than that of OM image. And the confirmation of the graphite boundary should influence directly the quantitative characterization of graphite morphology. In the process of defining the threshold for graphite rendering, area percentage of graphite in XRT image was deemed to be the same as that in OM image according to the comparative analysis of them. And thus the accuracy and validity of graphite rendering can be guaranteed.

Figure 3. 2D morphology of graphite in different cast iron observed by OM
(a) gray iron (b) CGI with 52.8% vermicularity
(c) CGI with 76.1% vermicularity (d) CGI with 91.3% vermicularity
Fig. 3 shows the 2D OM images of the other cast irons studied in present research which can be used as the analytical comparison. The 2D graphite morphology in gray iron exhibits typical thin flaky character, as shown in Fig.3a. As for CGI, the quantity of vermicular graphite increases while the quantity of spherical graphite decreases with the increase of vermicularity. From 2D perspective the graphite morphology in CGI with 52.8% vermicularity (Fig.2b) is similar to that of ductile iron (Fig.1a). The morphology of vermicular graphite and flake graphite is obviously different, especially from the perspective of the width and head shape of graphite. In summary, the graphite 2D morphology of the samples which is used to analyze the 3D morphology quantitatively is very representative.

![Graphite Morphology](image)

**Figure 4.** The spatial structure and morphology of graphite in different cast iron
(a)(b) gray iron, (c) ductile iron, (d) CGI with 76.1% vermicularity

Fig. 4 shows the spatial structure and 3D morphology of graphite in different cast iron. Different color rendering represents the size grades of isolated graphite clusters. There are obvious differences in graphite 3D morphology between three kinds of cast iron. As shown in Fig.4c, the nodular graphite in ductile iron is basically isolated. There are also isolated nodular graphite in CGI with 76.1% vermicularity. But a considerable amount of graphite interconnected into some large graphite clusters, as shown in Fig.4d. As for gray iron, almost all graphite phases are interconnected, as shown in Fig.4a. The other little isolated graphite truncated by the boundary of the unit cell is also probably connected in a larger unit cell. In addition, the flake-shaped graphite in gray iron shown in Fig.4b is obviously different from coral-shaped graphite in CGI. The interconnectivity of large flake graphite in gray iron is more evident.

Fig. 5 shows the spatial structure and morphology of graphite in CGI with different vermicularity (52.8%, 66.5%, 83.9% and 91.3% respectively). When the vermicularity is low, for example, 52.8% and 66.5%, graphite exhibits spherical and stubby shape. With the increase of vermicularity, the cross-sectional thickness of graphite is getting thinner and thinner. The cross-section morphology of CGI with 91.3% vermicularity represents the flake graphite feature roughly. And the dimension of the
largest graphite cluster is getting bigger. The network connectivity characteristics of graphite is more obvious. Especially for 91.3% vermicularity, the largest graphite cluster almost runs through the whole unit cell.

![Figure 5](image)

**Figure 5.** The spatial structure and morphology of graphite in CGI with different vermicularity (a)52.8% (b)66.5% (c)83.9% (d)91.3%

### 3.2 Quantitative Characterization of Graphite in Cast Iron

In order to further characterize the quantitative difference of graphite morphology among different cast iron samples, the distribution of graphite size is discussed according to the graphite cluster’s volume. The number of graphite clusters within each 10000μm³ interval is counted and shown in Fig.6. The number of small size graphite clusters is very large and the proportion of graphite clusters smaller than 10000μm³ in different cast iron is listed in Table 1. Especially in gray iron and CGI with 91.3% vermicularity, this distribution characteristic of graphite cluster’s volume is more obvious and their proportion is as high as 99.7% and 97.1% respectively. In comparison, the proportion of graphite clusters smaller than 10000μm³ is only 39.5% and 35.5% respectively in ductile iron and CGI with 52.8% vermicularity. And the proportion increases gradually with the increase of vermicularity. In addition, the number of graphite clusters larger than 100000μm³ is quite few and generally less than 1 within each 10000μm³ interval. These data indicate that the graphite cluster’s volume is divided into two extremes in gray iron and CGI with high vermicularity. By contrast, the graphite cluster’s volume is relatively uniform in ductile iron and CGI with low vermicularity.

| Table 1 | The proportion of graphite clusters smaller than 10000μm³ in different cast iron |
|---------|---------------------------------------------------------------------------------|
|         | gray iron | ductile iron | CGI with different vermicularity / % | 52.8 | 66.5 | 76.1 | 83.9 | 91.3 |
| the proportion / % | 99.7       | 39.5         | 35.5 | 36.4 | 63.9 | 89.6 | 97.1 |
Figure 6. The distribution of graphite cluster’s volume in different cast iron samples

In Fig.6 most of the statistics are concentrated in the volume range from 10000 to 100000μm$^3$, as marked in a circle. Statistics in the volume range from 10000 to 100000μm$^3$ is extracted in order to distinguish these curves clearly, as shown in Fig.7. The number of graphite clusters within each 10000μm$^3$ interval gradually decreases with the increase of the volume of the graphite cluster. Compared with gray iron and CGI with high vermicularity, this downward trend is apparently slow in ductile iron and CGI with low vermicularity. With the exception of CGI with 52.8% vermicularity, this downward trend gradually accelerates with the increase of vermicularity in CGI. The difference of graphite volume distribution among different cast iron is mainly concentrated in the volume range from 10000 to 100000μm$^3$.

Figure 7. The distribution of graphite cluster’s volume ranged from 10000 to 100000μm$^3$ in different cast iron samples

In comparison, the graphite clusters larger than 100000μm$^3$ is very few in number, as shown in Fig.8. Only one graphite cluster is larger than 100000μm$^3$ in gray iron. The number of graphite clusters larger than 100000μm$^3$ is only 2 and 4 in ductile iron and CGI with 93.1% vermicularity respectively. As for CGI, the number of graphite clusters larger than 100000μm$^3$ decreases with the increase of vermicularity. There are 114 graphite clusters larger than 100000μm$^3$ in CGI with 52.8% vermicularity. Considered from this point, ductile iron is quite different from CGI with low vermicularity.
The number of graphite clusters larger than 100000μm$^3$ in different cast iron samples (percentage represents vermicularity of CGI)

Fig. 8. The number of graphite clusters larger than 100000μm$^3$ in different cast iron samples (percentage represents vermicularity of CGI)

Fig. 9 shows the total volume of graphite clusters larger than 100000μm$^3$ in different cast irons. As for gray iron and CGI, the total volume of graphite clusters larger than 1000000μm$^3$ basically accounts for most of graphite volume, although their number is very few. With the increase of vermicularity, the volume proportion of graphite clusters larger than 100000μm$^3$ basically increases. Especially in gray iron and CGI with high vermicularity, its volume fraction is over 95%. By contrast, the total volume of graphite clusters larger than 100000μm$^3$ is very small and its volume fraction is only 0.7% in ductile iron.

Fig. 9. The total volume of graphite clusters larger than 100000μm$^3$ in different cast iron samples (percentage represents vermicularity of CGI)

4. Conclusion
In the present study, based on 2D metallographic photos, X-ray tomography was utilized to investigate 3D morphology of graphite clusters in different cast iron and 3D quantitative characterization of graphite morphology was carried out by Avizo software. In order to further characterize the quantitative difference of graphite morphology among different cast iron samples, the distribution of graphite size is discussed according to the graphite cluster’s volume. Some conclusions can be drawn as follows.

1) The confirmation of the graphite boundary during the rendering of the graphite in Avizo software should influence directly the quantitative characterization of graphite morphology. Comparative analysis between XRT image and OM image can improve the accuracy and validity of graphite rendering.
2) The nodular graphite in ductile iron is basically isolated and almost all graphite phases are interconnected in gray iron. The interconnectivity of compacted graphite iron is closely related with vermicularity. The flake-shaped graphite in gray iron is obviously different with coral-shaped graphite in compacted graphite iron from 3D perspective.

3) No matter the number or the total volume of graphite clusters larger than 100000μm³, ductile iron is quite different from CGI with low vermicularity.

4) The quantity proportion of graphite clusters smaller than 10000μm³ is obviously the highest, especially, that in gray iron and CGI with 91.3% vermicularity is as high as 99.7% and 97.1 respectively. The volume proportion of graphite clusters larger than 100000μm³ basically accounts for most of graphite volume and is over 95% in gray iron and CGI with high vermicularity. The difference of graphite volume distribution among different cast iron is mainly concentrated in the volume range from 10000 to 100000μm³.

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5. Reference
[1] S. Dawson, Compacted graphite iron – a material solution for modern diesel engine cylinder blocks and heads, China Foundry, 6(2009), pp. 241-246
[2] V. Norman, P. Skoglund, D. Leidermark, J. Moverare, Thermo-mechanical and superimposed high-cycle fatigue interactions in compacted graphite iron, International Journal of Fatigue, 80(2015), pp. 381-390
[3] E. Hosseini, S.R. Holdsworth, Cracking due to combined TMF and HCF loading in cast iron, International Journal of Fatigue, 99(2017), pp. 279-285
[4] D. Holmgren, Review of thermal conductivity of cast iron, International Journal of Cast Metal Research, 18(2005), pp. 331-345
[5] D. Holmgren, A. Dioszegi, I.L. Svensson. Effects of nodularity on thermal conductivity of cast iron, International Journal of Cast Metal Research, 20(2007), pp. 30-41
[6] D. Holmgren, R. Kallbom, I.L. Svensson, Influences of the graphite growth direction on the thermal conductivity of cast iron, Metallurgical and Materials Transaction A, 38(2007), pp. 268-275
[7] M. Selin, D. Holmgren, I.L. Svensson, Influence of alloying additions on microstructure and thermal properties in compact graphite irons, International Journal of Cast Metal Research, 22(2009), pp. 283-285
[8] A. Velichko, A. Wiegmann, F. Mücklich. Estimation of the effective conductivities of complex cast iron microstructures using FIB-tomographic analysis, Acta Materialia, 57(2009), pp. 5023-5035
[9] R.L. Hecht, R.B. Dinwiddie, H. Wang. The effect of graphite flake morphology on the thermal diffusivity of gray cast irons used for automotive brake discs, Journal of Materials Science, 34(1999), pp. 4775-4781
[10] J.C. Pina, V.G. Kouznetsova, M.G.D. Geers, Thermo-mechanical analysis of heterogeneous materials with a strongly anisotropic phase: the case of cast iron, International Journal of Solids and Structures, 63(2015), pp. 153-166
[11] J.C. Pina, S. Shafiqat, V.G. Kouznetsova, J.P.M. Hoe芬agels, M.G.D. Geers. Microstructural Study of the Mechanical Response of Compacted Graphite Iron: An Experimental and Numerical Approach. Materials Science and Engineering A, 658(2016), pp. 439-449
[12] A. Velichko, C. Holzapfel, A. Siefers, K. Schladitz, F. Mücklich, Unambiguous classification of complex microstructures by their three-dimensinal parameters applied to graphite in cast iron. Acta Materialia, 56(2008), pp. 1981-1990
[13] M. Metzger, T. Seifert, Computational assessment of the microstructure-dependent plasticity of lamellar gray cast iron – part I: methods and microstructure-based models, International Journal of Solids and Structures, 66(2015), pp. 184-193
[14] G. Fischer, J. Nellesen, N.B. Anar, K. Ehrg, H. Riesemeier, W. Tillmann, 3D analysis of
micro-deformation in VHCF-load nodular cast iron by μCT, Materials Science and Engineering A, 577(2013), pp. 202-209
[15] J.M. Li, L. Lu, M.O. Lai, Quantitative analysis of the irregularity of graphite nodules in cast iron, Materials Characterization, 45(2000), pp. 83-88
[16] A. Velichko, C. Holzapfel, F. Mücklich, 3D characterization of graphite morphologies in cast iron, Advanced Engineering Materials, 9(2007), pp. 39-45
[17] A. Hatton, M. Engstler, P. Leibenguth, F. Mücklich, Characterization of graphite crystal structure and growth mechanisms using FIB and 3D image analysis, Advanced Engineering Materials, 13(2011), pp. 136-144
[18] V. D. Cocco, D. Iacoviello, F. Iacoviello, A. Rossi, Graphite nodules influence on DCIs mechanical properties: experimental and numerical investigation, Procedia Engineering, 109(2015), pp. 135-143
[19] C. Chuang, D. Singh, P. Kenesei, J. Almer, J. Hryn, R. Huff, 3D quantitative analysis of graphite morphology in high strength cast iron by high-energy X-ray tomography, Scripta Materialia, 106(2015), pp. 5-8
[20] S.R. Stock, X-ray microtomography of materials, International Materials Reviews, 44(1999), pp. 141-164
[21] Y.Z. Liu, Y.F. Li, J.D. Xing, S.G. Wang, B.C. Zheng, D. Tao, W. Li, Effect of graphite morphology on the tensile strength and thermal conductivity of cast iron, Materials Characterization, 144(2018), pp. 155-165
[22] Y. Wu, J.P. Li, Z. Yang, Y.C. Guo, Z.J. Ma, M.X. Liang, T. Yang, D. Tao, Creep behavior of a high strength compacted graphite cast iron, Chinese Journal of Materials Research, 33(2018), pp. 43-52