Materials Science & Engineering A 664 (2016) 75–85

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

Influence of thermal exposure on microstructure evolution and tensile fracture behaviors of compacted graphite iron

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ARTICLE INFO

Article history:
Received 2 February 2016
Received in revised form
28 March 2016
Accepted 28 March 2016
Available online 29 March 2016

Keywords:
Compacted graphite iron
Thermal exposure
In-situ tensile testing
Microstructure evolution
Fracture mechanism

ABSTRACT

The mechanical properties of compacted graphite iron (CGI) may undergo degradation when it exposes at high temperature environment for a long period of time. In this study, the influences of thermal exposure conditions on microstructure evolution, tensile properties, especially in-situ tensile fracture behaviors of CGI were investigated. The experimental results show that the cementite surrounding the vermicular graphite (VG) gradually decomposes into ferrite and graphite with increasing the thermal exposure time. The increase of ferrite content with lower strength but higher ductility around VG particles, and the size and content of VG particles can lead to the easier initiation of crack, decrease of strain hardening rate, and the retard of the crack propagation rate in the matrix. All these factors often cause the decrease of strength but increase of elongation to fracture for CGI. Finally the relationship among yield strengths of CGI and matrix, and graphite content was discussed.

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1. Introduction

Compacted graphite iron (CGI) is an important engineering material which was firstly studied in 1948 [1]. Vermicular graphite (VG) particles presented in CGI are in short, stubby, wormlike shape with round edges in two dimensions (2D); however, they are connected in three dimensions (3D) [2]. These special morphologies often result in a good combination of thermal conductivity and mechanical properties [3–6]. Therefore, CGI has been widely applied in modern industries, especially in the diesel cylinder head [7,8].

Cylinder head always serves at elevated temperatures, so their microstructures may undergo degradation. From its failure analysis, Xu et al. [9] observed pearlite decomposition. Ghodrat et al. [10] also noticed the same phenomenon at temperature above 420 °C due to the volume expansion of CGI. Kim et al. [11] found that the weight loss at 500 °C increased rapidly for CGI with more than 70% pearlite comparing with it at 200 °C and 300 °C. This may deteriorate the mechanical properties of CGI in diesel components according to the similar tests in the previous study [12]. However, the effect of microstructure evolution in CGI on its mechanical properties under working conditions has been seldom investigated systemically. For a better understanding of the corresponding mechanism, it is necessary to apply the thermal exposure technique to simulate its working condition at elevated temperatures.

With development of techniques, more advanced equipment has been applied to investigate the microstructures and tensile fracture behaviors of cast irons. Alexandra et al. and Hatton et al. [13–15] observed the microstructure evolution of the VG by focused ion beam (FIB) and transmission electron microscope (TEM). They confirmed the assumption of crystallographic structure made by Llorca-Isern et al. [16] and Tartera et al. [17] that VG grows initially with a near-spherical shape. Chuang et al. [2] utilized high-energy synchrotron X-ray tomography to perform quantitative 3D-characterization of the distribution of graphite particles in the high-strength CGI as mentioned above. After research on the tensile fracture behaviors of spherical graphite iron (SGI) and lamellar graphite iron (LGI) using in-situ technique with scanning electron microscope (SEM), it was observed that the yielding appearance of cast irons was either caused by the initiation of slip bands in the matrix [18,19] or micro-cracks between matrix and graphite [20,21]. Comparing with other techniques, in-situ SEM technique may provide an opportunity for researchers to reveal the tensile fracture behaviors directly, and may help understand the fracture mechanisms deeply. For example, Cocco et al. [5,22] directly observed the tensile fracture behaviors of SGI which were divided into five steps by other researchers [23–26], and summarized the debonding mechanisms of graphite, i.e. “onion-like” mechanism and “disgregation” mechanism. Since in-situ technique

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http://dx.doi.org/10.1016/j.msea.2016.03.118
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using SEM is very helpful to investigate the fracture mechanisms of SGI and LGI, it should also supply a strong support for the investigation on CGI.

In the present study, the influence of thermal exposure on the microstructure evolution, tensile properties, fracture behaviors and corresponding mechanisms of CGI were investigated using in-situ technique with SEM. Especially, the relationship among yield strengths of CGI, strength of matrix, the graphite content was established.

2. Experimental materials and procedures

Casted RuT300 (Chinese designation of CGI) was chosen in the present study. Its chemical composition is shown in Table 1. Thermal exposure technologies applied in the investigation are shown in Table 2. The dimension of rectangular ingot is about 100 mm × 30 mm × 120 mm. It was normalized at 900 °C for 1.5 h firstly and cooled to room temperature in air. After that, it was cut into three pieces with electric discharge machining. One of them without further heat treatment, and all the specimens from it were named as E0. The other two pieces were used to thermal exposure treatment. The exposure temperature was at 700 °C which should accelerate microstructure evolution [10]. The thermal exposure times were 4 h and 8 h, and the specimens from the corresponding pieces were named as E4 and E8, respectively.

In order to avoid the effect of oxidation behavior [10], tested specimens in the present study were cut from the inner part of each piece with electric discharge machining, then polished with abrasive and pastes. Graphite in the non-etched metallographic specimens was firstly observed with an Olympus optical microscope (OM). Area percentage of graphite, nodularity, mean aspect ratio [27] and average count of graphite particles in per square millimeter [28] were measured with image pro plus (IPP) that was based on the measurement of pixels' areas in the selected gray level range [29]. The area percentages of pearlite and ferrite were measured by comparing the OM images of the specimens etched by 4% nital solution with standard images. For more details of the microstructure, the further observations were conducted with secondary electron (SE) and back-scattered electrons (BSE) of SEM JSM6510. The former manifests the surface morphology of specimens, and the latter can provide the distribution of different particles identified by chemical elements. Meanwhile, micro hardness of matrix in the etched metallographic specimens was measured with Leco AMH-43 automatic hardness testing system. More than eight points located in the matrix were indented in each specimen, and the load was 0.1 kgf for 13 s.

The shapes and sizes of conventional and in-situ tensile specimens are shown in Fig. 1. The conventional tensile tests were conducted with an Instron 5982 universal testing machine at room temperature, and the fracture surfaces were observed with a JSM 6510 SEM. The conventional and in-situ tensile tests were carried out under a tensile strain rate of $5 \times 10^{-4}\,s^{-1}$ and a constant cross-head speed of 0.033 mm min^{-1}, respectively. And the tensile fracture behaviors were observed using an in-situ SEM tensile testing system assembled with SHIMADZU SEVEO 4830 and JSM 6510 SEM.

Table 1
The chemical composition of CGI (wt%).

CE	C	Si	Mn	P	S	Cu	Fe
4.4	3.65–3.8	2.1–2.4	0.65	≤ 0.05	≤ 0.03	0.4–0.8	Balance

Table 2
Treatment technologies of CGI.

No.	Treatment	Temperature (°C)	Time (h)
E0	Normalization	900	1.5
E4	Thermal exposure after normalization	700	4
E8	Thermal exposure after normalization	700	8

3. Experimental results

3.1. Microstructure evolution

The microstructures of CGIs with different thermal exposure times are shown in Figs. 2 and 3. In Fig. 2, it can be seen that CGIs mainly contains graphite, pearlite and ferrite, which are indicated with capital letters G, P and F, respectively. In Fig. 2(a), (c) and (e), the small gray worm-like spots are determined as VG particles, and the bright and dark parts in the matrix are pearlite zones and ferrite zones, respectively, i.e., the circles "c" in Fig. 2(c) and "e" in Fig. 2(e) indicate the pearlite zones, while the circles "d" in Fig. 2 (c) and "f" in Fig. 2(e) indicate ferrite zones. Especially, it was found there was no oxidation phenomenon in the microstructures of CGIs. Therefore, the effect of oxidation can be excluded. Notice that under the normalization condition, the pearlite zone and ferrite zone cannot be distinguished clearly because the area percentage of ferrite is very low. In Fig. 2(b), (d) and (f), graphite particles are black and in various shapes. Most of them are VG, and the others in circle like shape are nodular graphite (NG). Distributions of pearlite zones, ferrite zones, VG and NG particles are not uniform, especially, the particles distribute in clusters preferentially, i.e., the VG particles prefer to be embedded with ferrite zones, while most NG particles are embedded with pearlite zones (see Fig. 2(c) and (d)). Obviously, ferrite zones may spread until they connect with each other when the thermal exposure time increases. The area percentages of graphite, pearlite and ferrite, as well as the nodularity, mean aspect ratios and average counts of graphite for specimens E0, E4 and E8 are shown in Table 3. With increasing the thermal exposure time (see Fig. 2 and Table 3), the area percentage of pearlite decreases, but those of ferrite and graphite increase, and pearlite decomposes more quickly in the first 4 h. The mean aspect ratio of graphite becomes larger, while the average count becomes lager at first and then changes to smaller. For more details of pearlite decomposition, the selected areas (the zones in black and yellow dash circles) in Fig. 2 are re-amplified in Fig. 3. Fig. 3(a), (c) and (e) are the areas around NG particles, and Fig. 3 (b), (d) and (f) are that around VG particles. From Fig. 3, it can be seen that the cementites around NG particles in specimens E0, E4 and E8 are in laminar, short rod-like and granular shapes, respectively. Meanwhile, a few of residual granular cementites distribute around VG particles in E4 (Fig. 3(d)). Therefore, it can be inferred that pearlite decomposition is caused by the cementite breaking down and the pearlite around VG decomposes more quickly than that around NG. This is the reason why the VG particles are embedded with ferrite zones. Cementite is not stable and easily decomposed into ferrite and carbon at a certain high temperature according to the previous study [30]. When the carbon atoms exceed the solubility in ferrite during the cementite decomposition, they form graphite nuclei and diffuse into graphite particles. The nuclei will result in the increase of the average count of graphite particles in the early exposure times (see E4 in Table 3). Then coalescence of some graphite particles appear when they grow up by diffusion in a longer thermal exposure time. This will result in the decrease of average count. On the other hand, cementite decomposition rate is determined by specific surface area of graphite particles. The specific surface areas of VG particles are

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