Materials Science & Engineering A 654 (2016) 143–150

Contents lists available at ScienceDirect

Materials Science & Engineering A

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

Microstructure evolution and mechanical properties of drop-tube processed, rapidly solidified grey cast iron

Olamilekan Oloyede*, Timothy D. Bigg, Robert F. Cochrane, Andrew M. Mullis

Institute for Materials Research, School of Chemical and Process Engineering, University of Leeds, Leeds LS2 9JT, United Kingdom

ARTICLE INFO

Article history:
Received 25 August 2015
Received in revised form
27 November 2015
Accepted 11 December 2015
Available online 12 December 2015

Keywords:
Rapid solidification
Drop tube
Grey cast iron
Cooling rate
Microstructure
Microhardness

ABSTRACT

The microstructure, phase composition and microhardness of rapidly solidified grey cast iron BS1452 Grade 250 are compared against the conventionally solidified alloy. Powder samples were prepared using containerless processing via the drop-tube technique. The rapidly cooled droplets were collected and sieved into size range from $\geq 850\ \mu m$ to $\leq 53\ \mu m$ diameters corresponding to estimated rates of 200–23,000 K s^{-1}. Microstructure evaluations were made by optical and scanning electron microscopy, while XRD was used for identification and analysis of evolved phases. The control sample showed extensive graphite flake formation which was absent in virtually all the droplets samples. With decreasing particle size (increasing cooling rate) we observed an increase in the proportion of Fe_3C present and the retention of γ-Fe in preference to α-Fe, with the proportion of retained austenite increasing with increasing cooling rate. At the highest cooling rates utilised a Martensitic or acicular ferrite structure was observed. Cooling rates of 200 K s^{-1} resulted in a doubling of the measured microhardness relative to the as-received (slowly cooled) material. Cooling at the highest rates achieved resulted in a further doubling of the measured microhardness.

1. Introduction

Grey cast iron remains one of the most important casting materials, with over 70% of the total world's production tonnage [1]. The ultimate structure of this Fe–C–Si based alloy depends primarily on its composition and the cooling rate applied during shaping [2]. Generally, under 'normal' solidification conditions, slow cooling permits the formation of extensive flake graphite and as a result of this, the material may suffer from poor mechanical properties, particularly low ductility and a tendency towards brittle failure [3,4]. However, with recent developments in rapid solidification technology, methods such as containerless processing may be applied to control the cooling rate of the alloy for any given elemental composition [5]. This leads to modification of the morphology, size and distribution of the flake graphite and of the structure of the matrix, potentially giving rise to improved mechanical properties for the material [6].

Generally, cast irons are relatively high carbon alloys which are classified mainly as grey, ductile or white based upon the shape of graphite in grey and ductile irons and the existence of carbides in white irons. Graphite in grey iron appears as randomly distributed flakes but is nodular in ductile iron. Grey and ductile cast iron usually also contain some Si or other alloying additions, which act as stabilisers for the graphite, such that it precipitates out even at hypoeutectic compositions.

Grey cast iron, which is so called, not because of its colour, but due to the unreflective appearance of its fractured surface, is widely chosen in many industrial applications because of its flexibility in usage, good castability, low-cost (20–40% less than steel), corrosion resistance, machinability and low melting point. It also displays a high damping capacity due to its high carbon content, which gives it an advantage in modern automotive part manufacture. Conventional grey cast iron has a pearlite matrix and it is used extensively in the production of machine components such as disc brake rotors, gear bearings, engine casings and hydraulic valves [7–9].

Very limited investigations on the rapid solidification of grey cast iron have been undertaken to date. Behnam et al. [7] have studied the effect of cooling rate on primary dendrite arm spacing (DAS), secondary dendritic arm spacing (SDAS), thickness of ferrite–cementite layer (λ_e) and mechanical property (hardness) of grey cast iron. Their evaluation was that, as expected, higher cooling rates resulted in smaller DAS, SDAS and λ_e. The DAS was found to be related to the cooling rate, R, via a power law relationship with DAS$\propto R^{-0.61}$. λ_e was also found to follow a power law relationship, albeit with the smaller negative exponent of -0.16. These were found to correlate with hardness data via a

---

* Corresponding author.
E-mail addresses: pmoro@leeds.ac.uk (O. Oloyede), a.m.mullis@leeds.ac.uk (A.M. Mullis).

http://dx.doi.org/10.1016/j.msea.2015.12.020

quadratic relationship, with the hardness increasing by ~33% as the cooling rate was increased from 0 to 20 K s^{-1}. Another significant effort was made by Kiani-Rashid et al. [10], in which they outlined the effect of cooling rate on the formation of metastable phases in austempered nickel–molybdenum grey cast iron and how this affected its microhardness. Yang et al. [11], in a study of gas atomised grey cast iron powders, related the microstructural evolution of the powders to the particle size (and hence cooling rate). They showed that with increasing cooling rate there was a change in the flake morphology from random to oriented. Moreover, from their XRD analysis it was evident that with increasing cooling rate (decreasing particle size) the proportion of γ-Fe increased while that of α-Fe decreased. However, there was no evidence, either from their XRD or microstructural analysis, of any phases other than ferrite, austenite and Fe_3C.

In a study by Yi et al. [12] based on repair technology using the rapid solidification process of laser fusion welding, they discovered that overall crack toughness of the material can be increased around the repaired zone (RZ). This was further explained by Ebrahimnia et al. [13] who stated that cracks initiate mostly at the interface of graphite and then propagate through the matrix of a component thereby causing fracture. However they show that with laser processing this can be curtailed. As further established by Fu et al. [14], their results showed that the weldability of cast iron can be improved by laser cladding by inhibiting graphite precipitation at the interface between the weld zone and the preexisting material.

The purpose of this investigation is to produce rapidly solidified grey cast iron powders using a conventionally cast BS1452 Grade 250 ingot as the feedstock material. The microstructure and microhardness of the powders will then be assessed as a function of cooling rate and compared to that of the conventionally solidified feedstock material. Drop-tube processing is used for this purpose, it being a containerless processing technique by which rapid solidification of small droplets in-flight is achieved. As such it is an analogue for the commercial high pressure gas atomisation technique. This method is such that the cooling rate will increase in a predictable manner as the particle size decreases. Moreover, due to the increased rate of cooling, together with melt sub-division effects, the droplet undercooling will also increase with decreasing droplet size, albeit in a stochastic manner. This paper thereby presents results to show the dependence of the microhardness on the microstructure and phase evolution as a result of rapid solidification processing of grey cast iron.

2. Experimental procedure

Conventionally solidified, low alloy commercial grey cast iron to specification BS1452 Grade 250 was supplied as continuously cast 25 mmx1000 mm round bar by West Yorkshire Steel. Table 1 confirms the compositional analysis obtained for the alloy by XRF analysis and also gives the notional specification for grey cast iron, showing that all elemental constituents are within the range as prescribed by the ASM international standard [15]. The inoculant used in the supplied material was Supanoc (Si 70.51%; Al 1.64%; Ca 1.39%, balance Fe) which was added at the rate of 4 kg/T into the metal stream when pouring into the distribution ladle. The carbon equivalent (CE) value shows that the alloy is hypoeutectic, with its equivalent carbon content being less than 4.3%. The Calphad software package MTDATA was used to assess the equilibrium behaviour of the Fe–C–Si–P system with composition as given below, in order to confirm that the system was hypoeutectic, as per the simple equivalent carbon calculation.

Table 1
Elemental composition of commercial grey cast iron BS1452 grade 250 by XRF.

Element (wt%)	C	Si	Mn	P	S	Fe	CE
BS1452 Grade 250	2.70	2.83	0.58	0.15	0.054	93.34	3.70
ASTM A48 specification	2.5–4.0	1.0–3.0	0.2–1.0	0.002–1.0	0.02–0.025	96.28–90.96	Cal.

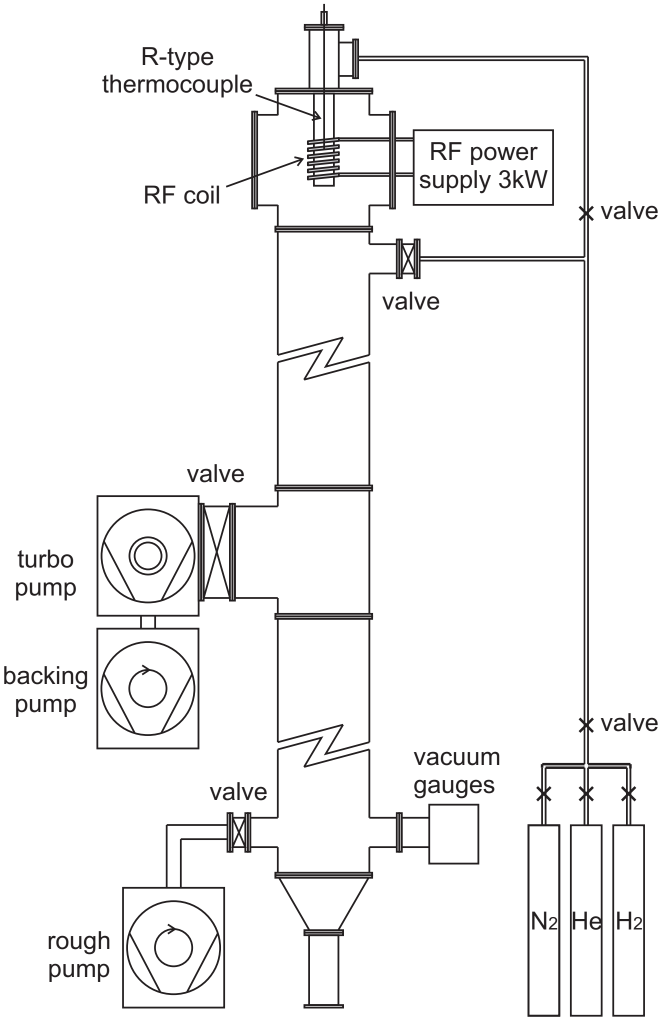

Fig. 1. Schematic diagram of the drop-tube apparatus used in this study.

This material was used as feedstock for the drop-tube experiments and also served as a reference material against which the effects of rapid solidification could be evaluated. To obtain rapidly solidified droplets from the bulk as-cast sample, small pieces weighing approximately 16 g were cut from the ingot and placed in an open-top alumina crucible with 3 laser drilled, 300 μm diameter, holes in the base. When the crucible is pressurised the melt is ejected through these holes to fall freely through the 6.5 m length of the drop-tube, wherein they undergo rapid cooling and solidify in-flight.

Fig. 1 shows a schematic illustration of the drop-tube apparatus used in this study. At the top of the drop-tube is the furnace used to melt the metal and produce the spray of droplets. The crucible sits inside a graphite susceptor which makes a pressure tight seal with the top flange on the tube, allowing it to be pressurised when the melt is to be ejected. The susceptor, which sits inside an alumina radiation shield, is induction heated using a 3 kW RF generator. Prior to melting the entire apparatus is evacuated and purged according to the following schedule: (i) firstly the tube is